**U.S. DEPARTMENT OF COMMERCE**
**National Technical Information Service**

PB-283 289

# Procedures Manual for Fabric Filter Evaluation

Southern Research Inst, Birmingham, Ala

**Prepared for**

Industrial Environmental Research Lab, Research Triangle Park, N C

Jun 78

United States
Environmental Protection
Agency

Industrial Environmental Research
Laboratory
Research Triangle Park NC 27711

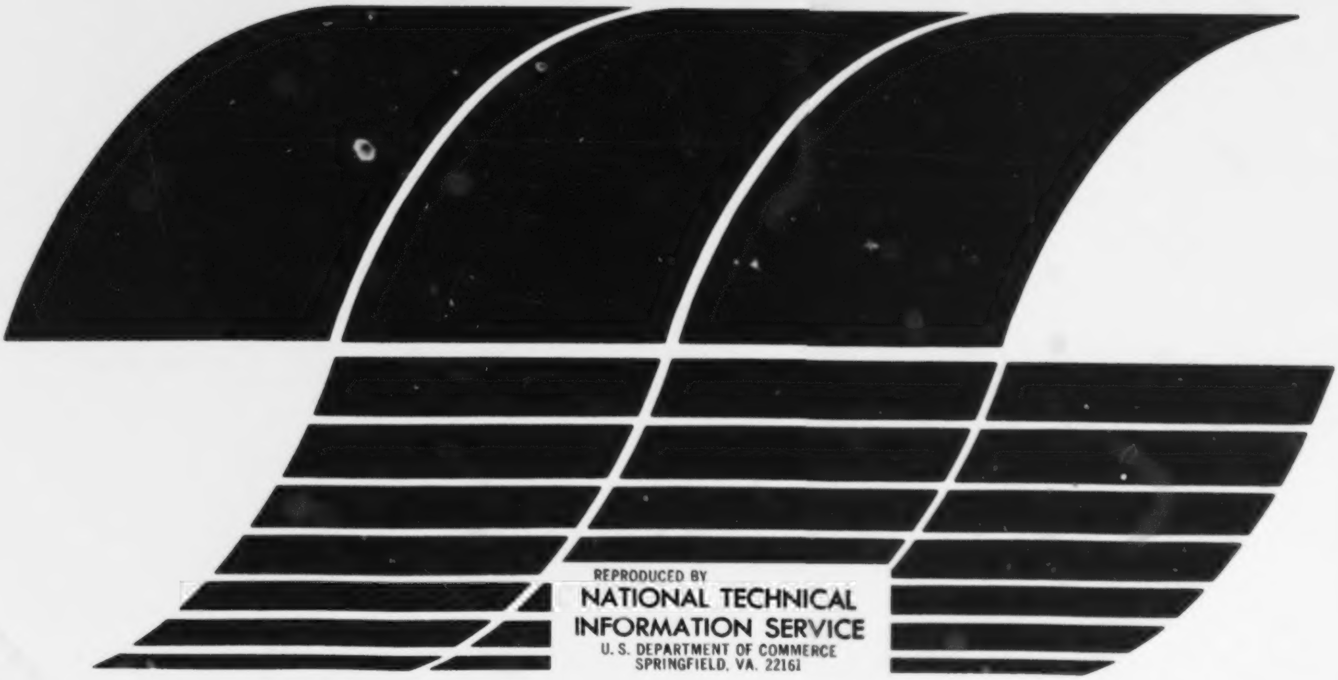
EPA-600/7-78-113
June 1978

PB 283 289

EPA

# Procedures Manual for Fabric Filter Evaluation

## Interagency Energy/Environment R&D Program Report



REPRODUCED BY
NATIONAL TECHNICAL
INFORMATION SERVICE
U. S. DEPARTMENT OF COMMERCE
SPRINGFIELD, VA. 22161

## RESEARCH REPORTING SERIES

Research reports of the Office of Research and Development, U.S. Environmental Protection Agency, have been grouped into nine series. These nine broad categories were established to facilitate further development and application of environmental technology. Elimination of traditional grouping was consciously planned to foster technology transfer and a maximum interface in related fields. The nine series are:

1. Environmental Health Effects Research
2. Environmental Protection Technology
3. Ecological Research
4. Environmental Monitoring
5. Socioeconomic Environmental Studies
6. Scientific and Technical Assessment Reports (STAR)
7. Interagency Energy-Environment Research and Development
8. "Special" Reports
9. Miscellaneous Reports

This report has been assigned to the INTERAGENCY ENERGY-ENVIRONMENT RESEARCH AND DEVELOPMENT series. Reports in this series result from the effort funded under the 17-agency Federal Energy/Environment Research and Development Program. These studies relate to EPA's mission to protect the public health and welfare from adverse effects of pollutants associated with energy systems. The goal of the Program is to assure the rapid development of domestic energy supplies in an environmentally-compatible manner by providing the necessary environmental data and control technology. Investigations include analyses of the transport of energy-related pollutants and their health and ecological effects; assessments of, and development of, control technologies for energy systems; and integrated assessments of a wide range of energy-related environmental issues.

## EPA REVIEW NOTICE

This report has been reviewed by the participating Federal Agencies, and approved for publication. Approval does not signify that the contents necessarily reflect the views and policies of the Government, nor does mention of trade names or commercial products constitute endorsement or recommendation for use.

This document is available to the public through the National Technical Information Service, Springfield, Virginia 22161.

## TECHNICAL REPORT DATA

*(Please read Instructions on the reverse before completing)*

| | | |
|---|---|---|
| 1. REPORT NO.<br>EPA-600/7-78-113 | 2. | 3. RECIPIENT'S ACCESSION NO.<br>PB283289 |
| 4. TITLE AND SUBTITLE<br>Procedures Manual for Fabric Filter Evaluation | | 5. REPORT DATE<br>June 1978 |
| | | 6. PERFORMING ORGANIZATION CODE |
| 7. AUTHOR(S)<br>Kenneth M. Cushing and Wallace B. Smith | | 8. PERFORMING ORGANIZATION REPORT NO.<br>SORI-EAS-78-355FFM |
| 9. PERFORMING ORGANIZATION NAME AND ADDRESS<br>Southern Research Institute<br>2000 Ninth Avenue, South<br>Birmingham, Alabama 35205 | | 10. PROGRAM ELEMENT NO.<br>EHE624 |
| | | 11. CONTRACT/GRANT NO.<br>68-02-2131, WA 21104 |
| 12. SPONSORING AGENCY NAME AND ADDRESS<br>EPA, Office of Research and Development<br>Industrial Environmental Research Laboratory<br>Research Triangle Park, NC 27711 | | 13. TYPE OF REPORT AND PERIOD COVERED<br>Task Final; 10/77-4/78 |
| | | 14. SPONSORING AGENCY CODE<br>EPA/600/13 |

15. SUPPLEMENTARY NOTES IERL-RTP project officer is D. Bruce Harris, Mail Drop 62, 919/541-2557.

16. ABSTRACT The report describes methods to be used in experimentally characterizing the performance of fabric filters for pollution control. It gives a detailed description of the mechanical characteristics of fabric filters. It describes procedures for measuring particle size distribution, the mass concentration of particulate matter, and the concentration of major gaseous components of the flue-gas/particle mixture. It gives a concise discussion and outline, describing the development of a test plan for evaluating a fabric filter installation. By following this outline, useful tests may be performed, ranging in complexity from qualitative and relatively inexpensive to rather elaborate research programs.

17. KEY WORDS AND DOCUMENT ANALYSIS

| a. DESCRIPTORS | b. IDENTIFIERS/OPEN ENDED TERMS | c. COSATI Field/Group |
|---|---|---|
| Air Pollution | Air Pollution Control | 13B |
| Dust Control | Stationary Sources | |
| Gas Filters | Particulate | 13K |
| Fabrics | Fabric Filters | 11E |
| Evaluation | Size Distribution | 14B |
| Measurement | Mass Concentration | |

| | | |
|---|---|---|
| 18. DISTRIBUTION STATEMENT<br>Unlimited | 19. SECURITY CLASS *(This Report)*<br>Unclassified | 21. NO. OF PAGES<br>454 |
| | 20. SECURITY CLASS *(This page)*<br>Unclassified | 22. PRICE<br>A20-A01 |

EPA Form 2220-1 (9-73)

ATTENTION

AS NOTED IN THE NTIS ANNOUNCEMENT, PORTIONS OF THIS REPORT ARE NOT LEGIBLE. HOWEVER, IT IS THE BEST REPRODUCTION AVAILABLE FROM THE COPY SENT TO NTIS.

EPA-600/7-78-113
June 1978

# Procedures Manual for Fabric Filter Evaluation

by

Kenneth M. Cushing and Wallace B. Smith

Southern Research Institute
2000 Ninth Avenue, South
Birmingham, Alabama 35205

Contract No. 68-02-2131
W.A. 21104
Program Element No. EHE624

EPA Project Officer: D. Bruce Harris

Industrial Environmental Research Laboratory
Office of Energy, Minerals, and Industry
Research Triangle Park, NC 27711

Prepared for

U.S. ENVIRONMENTAL PROTECTION AGENCY
Office of Research and Development
Washington, DC 20460

i-b

## TABLE OF CONTENTS

Page

Abstract . . . . . . . . . . . . . . . . . . . . . . . . . x

Acknowledgements . . . . . . . . . . . . . . . . . . . . . xi

1. INTRODUCTION . . . . . . . . . . . . . . . . . . . . . 1
  1.1. FABRIC FILTER INSTALLATIONS. . . . . . . . . . . . 2
    1.1.1. Particle Filtering Mechanisms. . . . . . 2
    1.1.2. Factors Affecting Filter Performance . . 9
    1.1.3. Filter Fabrics . . . . . . . . . . . . . 19
    1.1.4. Types of Fabric Filters. . . . . . . . . 24

  1.2. PARTICULATE SAMPLING FOR FABRIC FILTER EVALUATION . . . . . . . . . . . . . . . . . . . . . 28
    1.2.1. General Considerations . . . . . . . . . 28
    1.2.2. Particulate Mass Measurements. . . . . . 31
    1.2.3. Particle Sizing Techniques . . . . . . . 32

2. TECHNICAL DISCUSSION . . . . . . . . . . . . . . . . 33
  2.1. MECHANICAL CHARACTERIZATION OF A FABRIC FILTER INSTALLATION. . . . . . . . . . . . . . . . . . 33
    2.1.1. Mechanical Design and Operating Data . . 34
    2.1.2. The Fabric Filter Bags . . . . . . . . . 35
    2.1.3. Filter Fabrics . . . . . . . . . . . . . 37
    2.1.4. Dust Removal Systems . . . . . . . . . . 39
    2.1.5. Baghouse Operation-General Maintenance Considerations. . . . . . . . . . . . . 39

  2.2. MASS EMISSION MEASUREMENTS . . . . . . . . . . . 46
    2.2.1. General Discussion . . . . . . . . . . . 46
    2.2.2. EPA-Type Particulate Sampling Train (Method 5). . . . . . . . . . . . . . 47
    2.2.3. ASTM-Type Particulate Sampling Train . . 49
    2.2.4. ASME-Type Particulate Sampling Train . . 49
    2.2.5. General Sampling Procedures. . . . . . . 51

  2.3. PARTICLE SIZE MEASUREMENT TECHNIQUES . . . . . . 54
    2.3.1. General Discussion . . . . . . . . . . . 54
    2.3.2. Inertial Particle Sizing Devices . . . . 56
    2.3.3. Optical Measurement Techniques . . . . . 73
    2.3.4. Ultrafine Particle Sizing Techniques . . 75

TABLE OF CONTENTS (Cont'd.)

Page

2.4. PROCESS EFFLUENT GAS ANALYSIS . . . . . . . . . . . . . 82
  2.4.1. General Discussion . . . . . . . . . . . . . 82
  2.4.2. Qualitative Gas Analysis. . . . . . . . . . . 83
  2.4.3. Quantitative Gas Analysis . . . . . . . . . . 83

3. DEVELOPMENT OF TEST PLANS FOR FABRIC FILTER EVALUATIONS . 87
3.1. OBJECTIVES OF CONTROL DEVICE TESTS. . . . . . . . . . 87
3.2. TYPE AND NUMBER OF TESTS REQUIRED . . . . . . . . . . 88
  3.2.1. Fabric Filter Level A Evaluation. . . . . . . 93
    3.2.1.1. Plant Operating Data. . . . . . . 93
    3.2.1.2. Baghouse-Fabric Filter Design Data . . . . . . . . . . . . . . . 94
    3.2.1.3. Flue Gas Characteristics, Baghouse ΔP, Maintenance Data . . . 94

  3.2.2. Fabric Filter Level B Evaluation. . . . . . . 98
    3.2.2.1. Quantitative Gas Analysis . . . . 98
    3.2.2.2. Inlet and Outlet Mass Concentration Measurements Total Mass Collection Efficiency. . . . . . 99

  3.2.3. Fabric Filter Level C Evaluation. . . . . . . 99

3.3. GENERAL PROBLEMS AND CONSIDERATIONS . . . . . . . . . 102

APPENDICES

Appendix A - AEROSOL FUNDAMENTALS, NOMENCLATURE, AND DEFINITIONS . . . . . . . . . . . . . . . . . . . . . . 107

Appendix B - PARTICULATE MASS CONCENTRATION MEASUREMENTS. . . 129

Appendix C - CASCADE IMPACTOR SAMPLING TECHNIQUES . . . . . . . 188

Appendix D - SIZE DISTRIBUTIONS OF SUBMICRON AEROSOL PARTICLES . . . . . . . . . . . . . . . . . . . . . 250

Appendix E - SUMMARY OF SOURCE PERFORMANCE METHODS. . . . . . . 302

Appendix F - FEDERAL STATIONARY SOURCE PERFORMANCE STANDARDS. 420

Bibliography. . . . . . . . . . . . . . . . . . . . . . . . 426

LIST OF FIGURES

Figure No. Page

1 Typical air flow--fabric filter configurations . . . . . . . . . . . . . . . . . . . . . . 3

2 Baghouse air flow and collection diagram . . . . 4

3 Typical simple fabric filter baghouse design . . 5

4 Typical pulse-jet baghouse with screw conveyor dust removal system . . . . . . . . . . . . . . 6

5 Types of dust filtration . . . . . . . . . . . . 8

6 Resistance changes during fly ash filtration for well-used fabrics. . . . . . . . . . . . . 10

7 Calculated effluent concentration vs. time for fly ash and ambient air dust based on measurements with an optical particle counter. . . . . 13

8 Effluent concentration vs. filter fabric loading . . . . . . . . . . . . . . . . . . . . . 14

9 Effluent concentration vs. filter fabric loading and particle size. . . . . . . . . . . . . 15

10 Penetration as a function of particle diameter and coal-fired boiler load. . . . . . . . . . . 16

11 Average outlet mass concentration (with one standard deviation limits) as a function of air-to-cloth ratio. . . . . . . . . . . . . . . . . 17

12 Penetration (with one standard deviation limits) as a function of air-to-cloth ratio . . . . . . 18

13 Typical filter cloth weaves. . . . . . . . . . . 21

14 Basic fabric weaves used for woven filter bags . 22

15 Residual dust left after several periods of shaking of a fabric filter bag. . . . . . . . . 25

16 Reverse air flexing to clean dust collector bags by repressuring . . . . . . . . . . . . . . . . 26

LIST OF FIGURES (Cont'd.)

| Figure No. | | Page |
|---|---|---|
| 17 | Typical reverse--pulse baghouse during cleaning cycle. | 27 |
| 18 | Cleaning methods for filter bags | 29 |
| 19 | Types of fabric filter systems depending on cleaning method | 30 |
| 20 | EPA Method 5 Particulate Sampling Train. | 48 |
| 21 | ASTM Type Particulate Sampling Train | 50 |
| 22 | Typical gas velocity distribution at the inlet to a baghouse | 53 |
| 23 | Operation principle and typical performance for a cascade impactor. | 58 |
| 24 | Andersen Mark III Stack Sampler. | 60 |
| 25 | Modified Brink Model BMS-11 Cascade Impactor | 61 |
| 26 | MRI Model 1502 Inertial Cascade Impactor | 62 |
| 27 | Sierra Model 226 Cascade Impactor. | 63 |
| 28 | University of Washington Mark III Source Test Cascade Impactor. | 64 |
| 29 | Schematic of the Source Assessment Sampling System. | 67 |
| 30 | Three Stage Series Cyclone System. | 69 |
| 31 | Five Stage Series Cyclone System | 70 |
| 32 | Laboratory Calibration for the Five Stage Series Cyclone System | 71 |
| 33 | Operating principle for an optical particle counter | 74 |

LIST OF FIGURES (Cont'd.)

| Figure No. | | Page |
|---|---|---|
| 34a | Parallel plate diffusion battery. . . . . . . . | 77 |
| 34b | Parallel plate diffusion battery penetration curves for monodisperse aerosols (12 channels, 0.1 x 10 x 48 cm). . . . . . . . . . . . . . . | 77 |
| 35 | Screen type diffusion battery . . . . . . . . . | 78 |
| 36 | The electric mobility analyzer principle. . . . | 80 |
| 37 | Flow schematic and electronic block diagram of the Electrical Aerosol Analyser. . . . . . . . | 81 |
| 38 | Schematic diagram of apparatus for the collection of $SO_3$ by the condensation method . . . . | 85 |
| A1 | Examples of frequency or particle size distributions . . . . . . . . . . . . . . . . . . | 109 |
| A2 | A single particle size distribution presented in four ways . . . . . . . . . . . . . . . . . | 113 |
| A3 | Size distributions plotted on log probability paper. . . . . . . . . . . . . . . . . . . . | 114 |
| B1 | Thermocouple junction . . . . . . . . . . . . . | 131 |
| B2 | Standard pitot tube . . . . . . . . . . . . . . | 134 |
| B3 | S-type pitot tube . . . . . . . . . . . . . . . | 135 |
| B4 | Probe and nozzle assembly . . . . . . . . . . . | 139 |
| B5 | Set-up for calibration of dry gas meter and orifice meter. . . . . . . . . . . . . . . . . | 143 |
| B6 | Data sheet for calibrating dry gas meter and orifice meter. . . . . . . . . . . . . . . . . | 144 |
| B7 | Minimum number of traverse points per sample obtained from "Distances to Disturbances", upstream and downstream. . . . . . . . . . . . | 152 |
| B8 | Examples of equal area sample points. . . . . . | 154 |
| B9 | Percent water vapor in air at saturation. . . . | 159 |
| B10 | Percent water vapor with wet and dry bulb . . . | 160 |

LIST OF FIGURES (Cont'd.)

| Figure No. | | Page |
|---|---|---|
| B11 | Isokinetic flow patterns. . . . . . . . . . . . | 163 |
| B12 | Stack Sampling Nomograph (side 1) . . . . . . . | 166 |
| B13 | Stack Sampling Nomograph (side 2) . . . . . . . | 167 |
| C1 | Presurvey sampling with a cascade impactor . . | 189 |
| C2 | Typical sample train with a heated impactor . . | 193 |
| C3 | Nomograph for selecting nozzles for isokinetic sampling . . . . . . . . . . . . . . . . . . | 202 |
| C4 | Nomograph for sampling time selection (50 mg sample). . . . . . . . . . . . . . . . . . . | 207 |
| C5 | Hypothetical particle size distribution at a fabric filter outlet determined from Andersen impactor data. . . . . . . . . . . . . . . . | 245 |
| C6 | Hypothetical particle size distribution at a fabric filter outlet determined from Andersen impactor data. . . . . . . . . . . . . . . . | 246 |
| C7 | Hypothetical particle size distribution at a fabric filter inlet determined from Brink impactor data. . . . . . . . . . . . . . . . | 247 |
| C8 | Hypothetical fabric filter fractional efficiency based on the data presented in Figures C5 and C7 . . . . . . . . . . . . . . . . . . | 248 |
| D1 | Probe losses due to settling and diffusion for spherical particles having a density of 2.5 gm/cm$^3$ under conditions of laminar flow. . | 252 |
| D2 | Diffusional adsorption apparatus for removal of $H_2O$ from sample aerosol . . . . . . . . . . | 255 |
| D3 | Sample Extraction-Dilution System (SEDS). . . . | 258 |
| D4 | Sample Extraction Diluter, cut-away view. . . . | 260 |
| D5 | Calculated dilution versus true dilution for the Southern Research Institute Ultrafine Particle Diluter, 0.092 μm particles . . . . . | 261 |

## LIST OF FIGURES (Cont'd.)

Figure No. Page

D6 Calculated dilution versus true dilution for the Southern Research Institute Ultrafine Particle Diluter, 0.15 μm particles. . . . . . . 262

D7 Parallel Plate Diffusion Battery. . . . . . . . . . 265

D8 Screen type diffusion battery . . . . . . . . . . . 268

D9 Theoretical parallel plate diffusion battery penetration curves . . . . . . . . . . . . . . . . 285

D10 Schematic diagram of the electrical aerosol analyzer. . . . . . . . . . . . . . . . . . . . . . 286

D11 Hypothetical inlet size distribution at a fabric filter on a coal-fired boiler. . . . . . . . . . 300

## LIST OF TABLES

Table No. Page

I Summary of physical and chemical properties of industrial filter fibers. . . . . . . . . . . 23

II Type and frequency of fabric filter problems reported . . . . . . . . . . . . . . . . . . . . 43

III Commercial cascade impactor sampling system . . . 65

IV Particulate control device tasks. . . . . . . . . 91

V Three levels of effort for fabric filter evaluation . . . . . . . . . . . . . . . . . . . . . . 92

VI Maintenance Report - Type and Frequency of Problems . . . . . . . . . . . . . . . . . . . . 96

B1 Duct traversing length factors. . . . . . . . . . 153

B2 Example - Particulate source test data - ESP Inlet. . . . . . . . . . . . . . . . . . . 180

C1 Impactor decision making. . . . . . . . . . . . . 191

C2 Sampling information required . . . . . . . . . . 212

C3 Sample Calculation - Input Data and Results . . . 232

C4 Program flow. . . . . . . . . . . . . . . . . . . 249

D1 Diffusion battery sample calculation data - CNC . 275

D2 Program 1 - Particle Diffusivity (Df) . . . . . . 279

D3 Program 2 - Particle Penetration (p). . . . . . . 282

D4 EAA (Model 3030) Data Reduction Form. . . . . . . 291

D5 EAA Current Readings (I, in picoamps and Dilution Factors) . . . . . . . . . . . . . . . . . . . . 294

D6 EAA (Model 3030) Data Reduction Form. . . . . . . 297

D7 EAA (Model 3030) Data Reduction Form. . . . . . . 298

# ABSTRACT

The purpose of this procedures manual is to describe methods to be used in experimentally characterizing the performance of fabric filters for pollution control. A detailed description of the mechanical characteristics of fabric filters is presented. Procedures are described for measuring the particle size distribution, the mass concentration of particulate matter, and the concentration of major gaseous components of the flue gas-particle mixture. A concise discussion and outline is presented which describes the development of a test plan for the evaluation of a fabric filter installation. By following this outline useful tests may be performed which range in complexity from qualitative and relatively inexpensive to rather elaborate research programs.

# ACKNOWLEDGEMENTS

Members of the Environmental Engineering Division of Southern Research Institute who contributed to the preparation of this report include Mr. W. R. Dickson, Research Chemist and Miss Annette Duncan, Assistant Physicist.

We also appreciate the assistance and guidance of our Project Officer, Mr. D. Bruce Harris.

# 1. INTRODUCTION

Many different types of measurements must be made in order to accurately evaluate the performance of a fabric filter (baghouse)* installed to remove suspended particulate matter from an industrial process stream. Among the required measurements are determinations of the compositions of the gas and suspended particles and of the particles' concentration and size distribution. Also, the baghouse geometry and operating parameters must be recorded for proper interpretation of the measurements.

This document provides information and guidelines for use in planning and conducting tests to obtain the necessary data.

A brief description of fabric filters and various evaluation methods is provided in the remainder of this section. In Section 2 the methods of measuring the fabric filter operating parameters and the technical background and procedures for flue gas and particulate characterization are discussed. Section 3 describes the logic and procedures to be used in developing a test plan for the evaluation of a fabric filter installation. The Appendices contain detailed information on the test methods, as well as a listing of the Federal Stationary Source Performance Standards and Federal Stationary Source Testing Reference Methods.

---

*In this manual the terms fabric filter and baghouse are used interchangeably throughout the text.

## 1.1 FABRIC FILTER INSTALLATIONS

A fabric filter consists of a porous, flexible layer of textile material through which a dusty gas is passed to separate particles from the gas stream. Schematic representations of typical bag-air flow configurations are shown in Figures 1 and 2. As particles accumulate on the fabric, resistance to gas flow increases. Deposits are removed periodically by vigorous cleaning of the cloth to maintain the pressure drop across the filter within practical operating limits.

A baghouse is a large metal structure divided into two functional areas which holds the fabric filter bags. The gas inlet section may be part of the baghouse structure proper or it may be designed as a gas distribution manifold. The function of this dirty air plenum is to distribute evenly the gas to the fabric filter bags.

The gas outlet section is designed for the recombination of clean air exiting each of the fabric filter elements. The medium separating the clean and dirty gas areas is the fabric filter formed either as tubular bags or flat sheets. The hoppers that collect the particulate matter periodically dislodged from the fabric filters are usually designed as part of the dirty air plenum. Two typical baghouse designs are depicted in Figures 3 and 4.

### 1.1.1 Particle Filtering Mechanisms

Three distinct periods of fabric filter dust filtration can be identified (Koscianowski and Koscianowski, 1976). The start up phase is initiated with the unused fabric and is completed with the first cleaning of the filters. The transition phase begins when the filters are cleaned for the first time, and is completed when the remaining fabric dust load remains stabilized

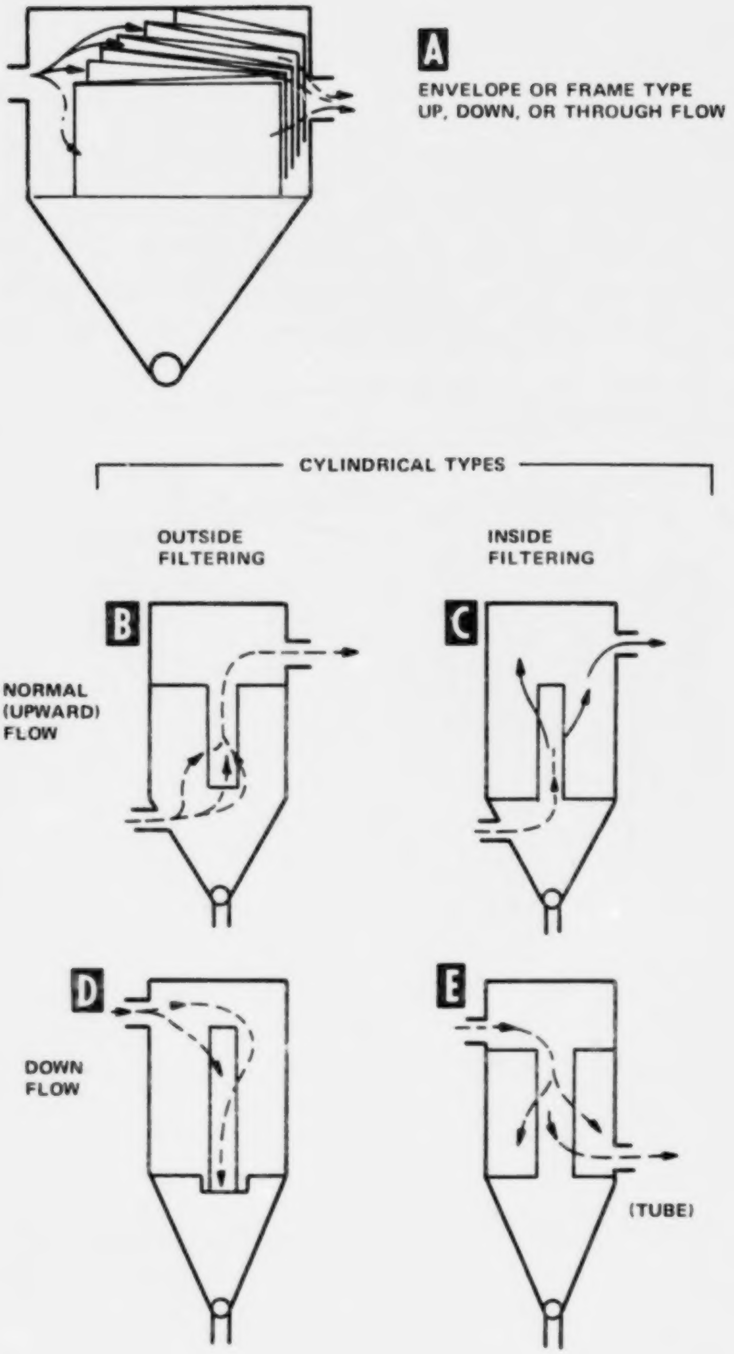

A
ENVELOPE OR FRAME TYPE
UP, DOWN, OR THROUGH FLOW
CYLINDRICAL TYPES
OUTSIDE FILTERING
INSIDE FILTERING
B
C
NORMAL (UPWARD) FLOW
D
E
DOWN FLOW
(TUBE)

*Figure 1. Typical air flow---fabric filter configurations.*

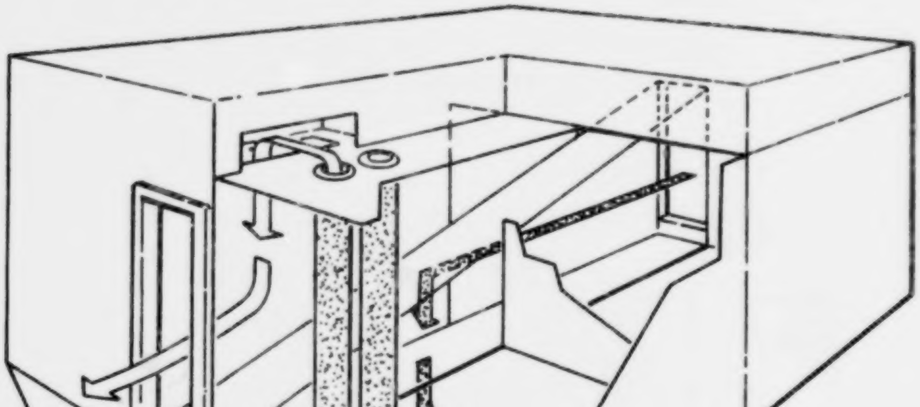


*Figure 2. Baghouse air flow and collection diagram.*

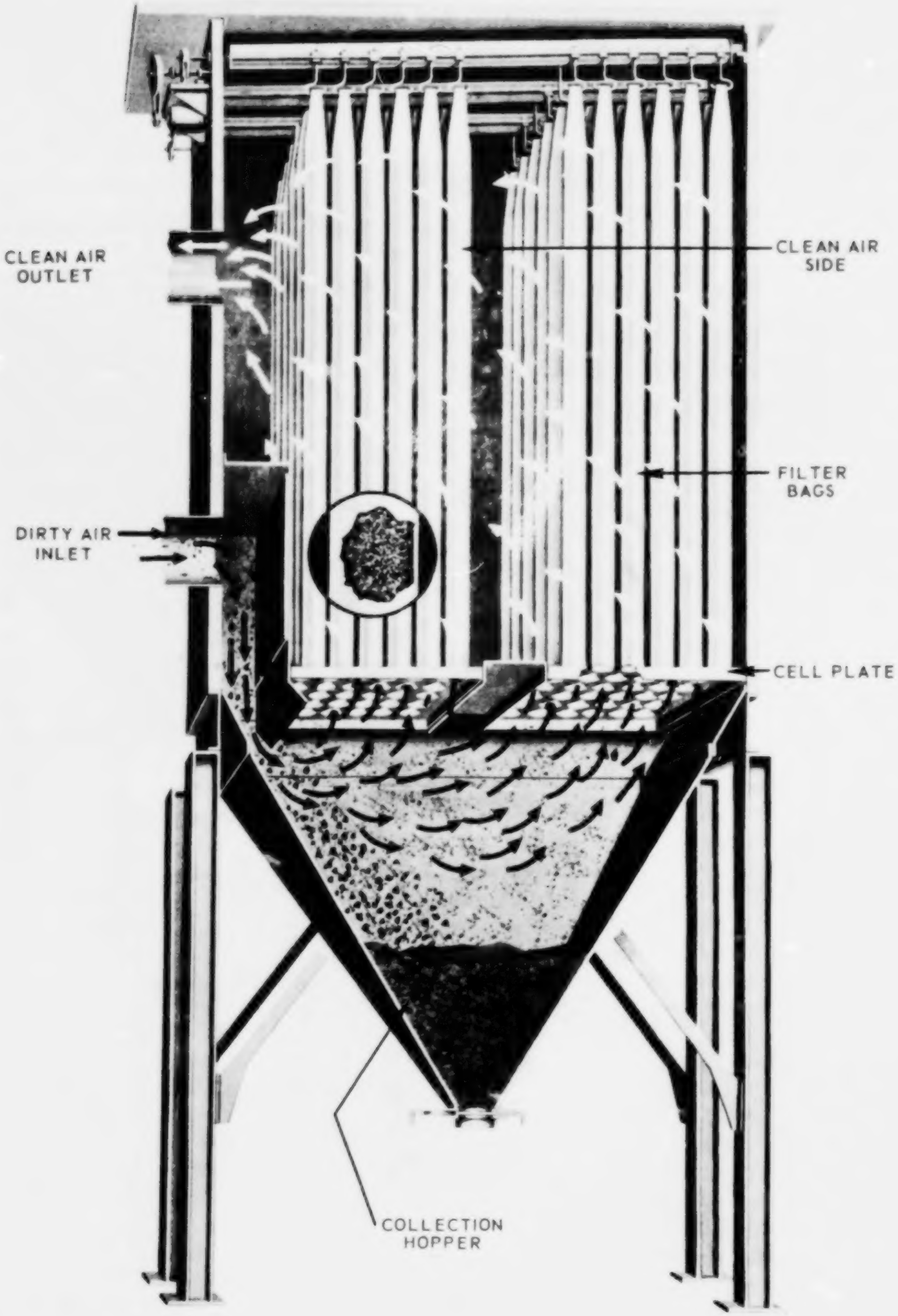

CLEAN AIR OUTLET
CLEAN AIR SIDE
FILTER BAGS
DIRTY AIR INLET
CELL PLATE
COLLECTION HOPPER

*Figure 3. Typical simple fabric filter baghouse design.*

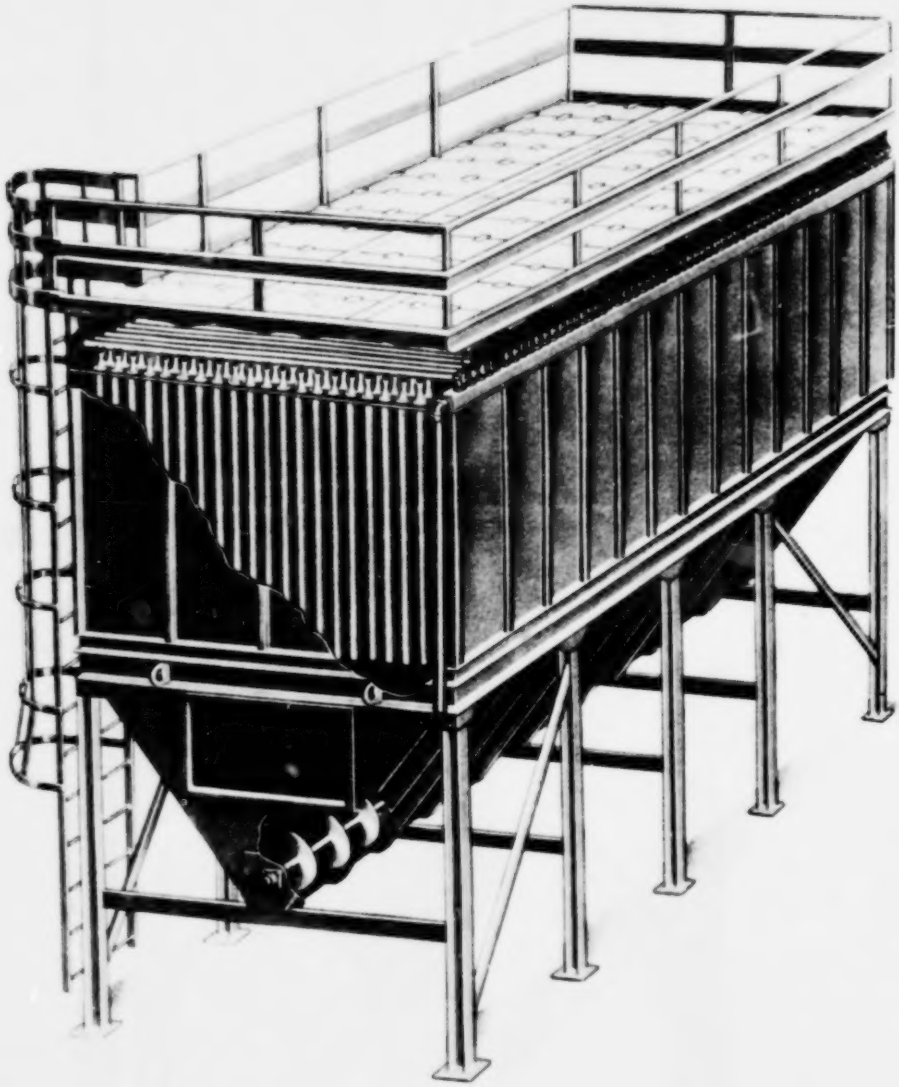


*Figure 4. Typical pulse-jet baghouse with screw conveyor dust removal system.*

after each cleaning cycle. The final phase is characterized by the fabric filters being filled with particulate matter and having a steady value of pressure drop and dust load immediately after each cleaning cycle. Figure 5 illustrates these three types of fabric filtering.

During the start-up phase of filtering the collection mechanisms of impaction, interception, and diffusion on and within the filter fibers operate (Dorman, 1966 and Billings and Wilder, 1970). Fabric materials woven from staple yarns (cotton, wool, etc.) capture particles by single projecting fibers within the air flow field, such as the interyarn spaces or pores. Smooth surface yarns (fiberglass and synthetic materials) have deep tunnel-like pores with few of these projecting fibers. Dust deposition occurs within these pores. Dust collected by the fabric filter builds up initially near these interyarn spaces. As it builds, a dust layer or cake is formed through which most of the flow must pass. This dust layer filtration continues until the pressure drop reaches a point where cleaning is initiated.

During the transition phase of filtration the residual dust load within the fabric accumulates. After each cleaning, the fabric is never returned to its unused condition since dust in at least a monolayer remains on the fibers. After each successive cleaning cycle, more dust is left in the fabric until this residual dust load remains constant. The length of time for this to occur is a function of several variables which include weave pattern, pore-size, air-to-cloth ratio (air flow divided by filter area), frequency of cleaning, method of cleaning and particle size distribution.

After the transition phase of filter life is complete, the final phase begins. At the beginning of each filtering cycle, the fabric pores are coated with dust. This residual dust plays an important part in the rapid formation of a new dust layer or

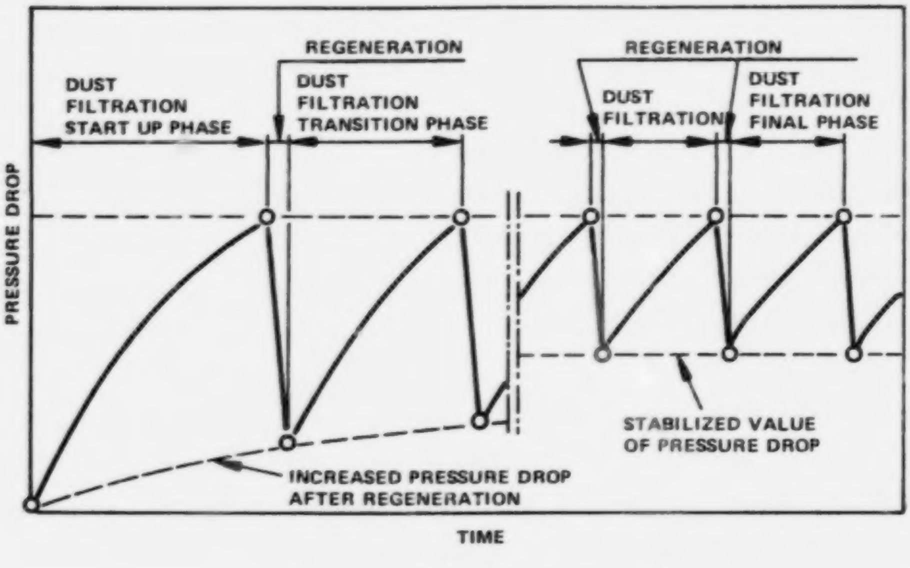


*Figure 5. Types of dust filtration.*

filter cake. Filtration begins with the capture of single particles by particles in the residual dust layer. These mechanisms are impaction, interception, and diffusion. These new particles act as obstructions and aid in the capture of still more particles. A series of particle aggregate chains project into the gas stream. Further accumulation occurs until the chains become long enough to bridge most of the interyarn spaces and the filter cake then is formed. During formation of the filter cake the pressure drop increases non-linearly with time. After the cake has been formed the pressure drop increases linearly with time (Billings and Wilder, 1970). Figure 6 shows this type of behavior for four different fabrics. This final phase filtering description applies to shake or reverse-air cleaning systems, but not to pulse-jet situations.

There is no comprehensive theory to describe the actual interrelationship or operation of these physical processes, however, Dennis, Cass, and Langley (1975) in their recent model studies of fabric filter collection assume that the flow and collection by the dust layer cake are like those in nuclepore and membrane filters with low porosity. Their model also makes the assumption that some of the interyarn pores always stay open during the cleaning and filtering cycles.

### 1.1.2 Factors Affecting Filter Performance

Several factors can influence fabric filter performance. These include fabric structure, air-to-cloth ratio, maximum pressure drop before cleaning, method of cleaning, cleaning frequency, intensity of cleaning, and flue gas temperature and humidity. These factors can be grouped into two areas. One is those affecting fabric filter design and the other is those factors which relate to the system as installed.

During the design phase questions of type of fabric, method of cleaning, air-to-cloth ratio, operating temperature and humidity should be considered. Once installed, the cleaning rate,

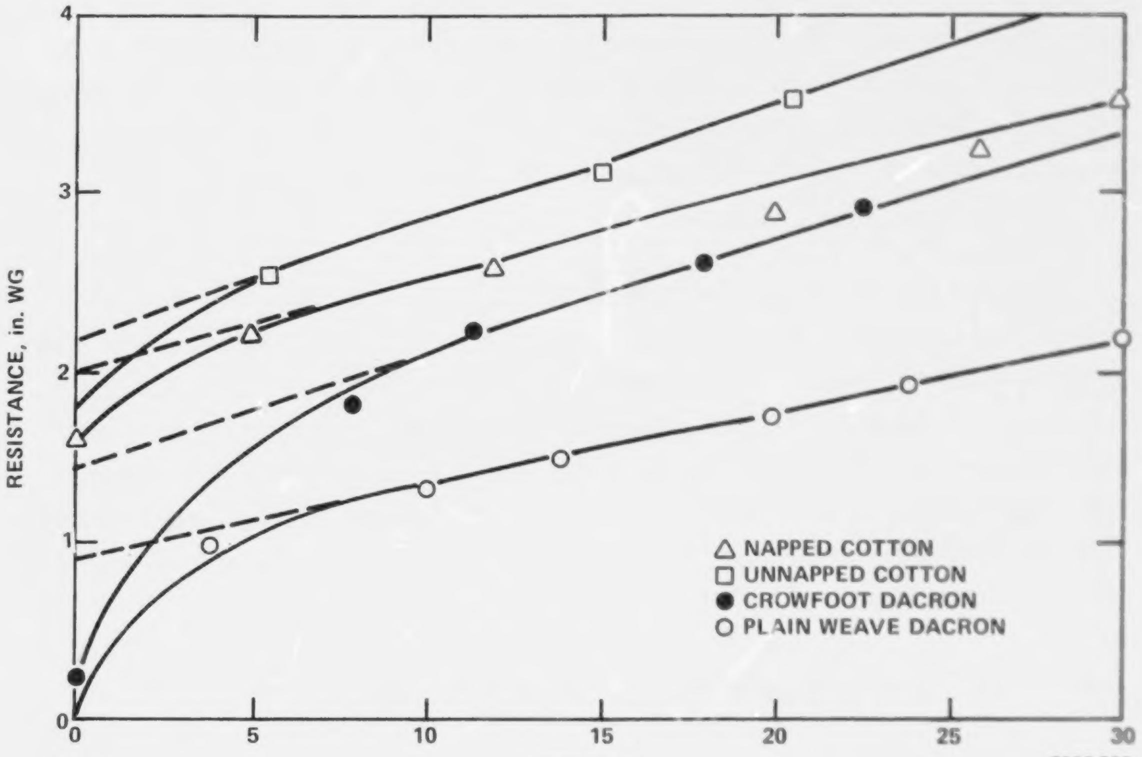


*Figure 6. Resistance changes during fly ash filtration for well-used fabrics. Inlet loading-3.5 gr/ft$^3$, filter velocity-3 ft/min, 30 min filter cycle. After Billings and Wilder(1970).*

duration, intensity of cleaning and other operating variables are adjusted to keep the system at peak efficiency.

The collection efficiency can also be affected by several other occurrences. Bags can be blinded (that is, clogged) during any filtering phase. Temperature excursions through acid dew points may cause condensation with resulting damage to the baghouse structure and adverse affects on the bag life and performance. Also, air preheater failures at utility boilers can cause temperature excursions above safe limits.

Fabric filter performance is not affected by some of the variables that are crucial for the success of scrubbers and electrostatic precipitators. Woven fabric filters are relatively insensitive to inlet dust loadings. Also, dust resistivity is of little consequence since electrostatic attraction is not the main method of removal. Particle wettability, necessary for good scrubber performance, is not important in fabric filters.

The insensitivity of fabric filters to inlet dust loading is well known (Billings and Wilder, 1970). A laboratory study (Dennis and Wilder, 1975) determined that the average mass emission and its size distribution may be almost independent of the loading and distribution of the inlet dust for a specified dust/ collector combination. The authors concluded that there was no simple relationship between typical inlet and outlet concentrations for most filter systems. This insensitivity of outlet loadings is understandable if a majority of the penetration is due to seepage. If this is the case, then the majority of emissions consist of previously deposited dust in the fabric.

The pressure drop across a baghouse is a factor in its performance. An operating pressure of 3-4 inches of water is typical for baghouses installed on utility boilers. Some industrial boilers, however, operate in excess of ten inches of water pressure drop. When filtration begins the pressure drop increases non-linearly until a cake is formed, after which it increases linearly with time. This behavior is illustrated in Figure 6.

Figure 7 shows that directly after a cleaning cycle, the filter efficiency drops to levels similar to those found in most precipitators (Dennis and Wilder, 1975). Penetration can either increase or decrease depending on the type of filter material, construction, and dust properties. Figures 8 and 9 illustrate two cases where penetration leveled off and remained fairly constant for seasoned fiberglass bags (Dennis, Cass, and Langley, 1975).

Fabric filter penetration increases rapidly for increasing air-to-cloth ratio or face velocity. Figures 10, 11, and 12 illustrate this performance characteristic for a utility boiler baghouse (Ensor, Hooper, and Scheck, 1976). Figure 10 shows baghouse penetration as a function of particle size and air-to-cloth ratio. Here the penetration of particles larger than 1.0 micrometer diameter increases with an increase in air-to-cloth ratio. This may be due to an increase in seepage of larger agglomerated particles. Such an increase has been measured during recent baghouse performance tests (Cass and Bradway, 1975). Figure 11 shows typical outlet mass concentrations as a function of boiler load. At lower air-to-cloth ratios, particles with larger diameters show a reduced penetration as shown in Figure 12.

The presence of pinhole inclusions in filter dust cakes can cause a degradation in performance. Pinholes are observed through collected dust surfaces, and occur for both woven and filtered fabrics (Holland and Rothwell, 1977). It has been thought that the increase in penetration in the 2 μm to 5 μm range of particle sizes is a direct result of pinhole leaks that allow coarser particles to pass through (Dennis, Cass, and Langley, 1975).

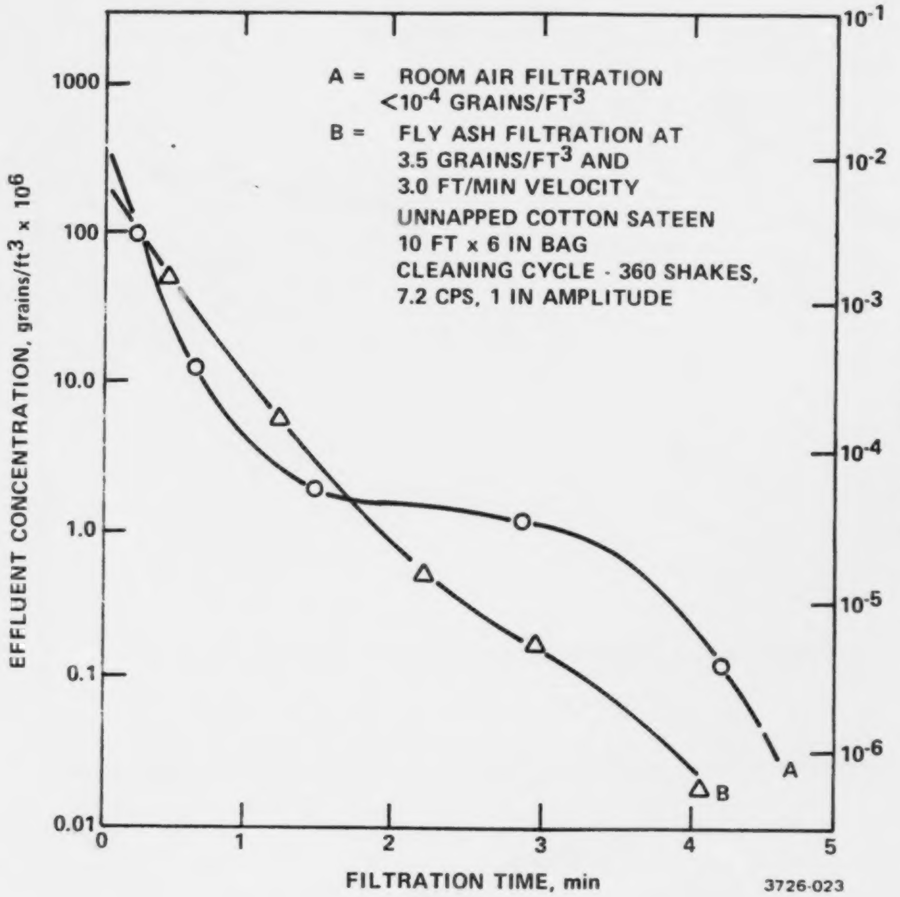


*Figure 7. Calculated effluent concentration vs. time for fly ash and ambient air dust based on measurements with an optical particle counter. After Billings and Wilder(1970).*

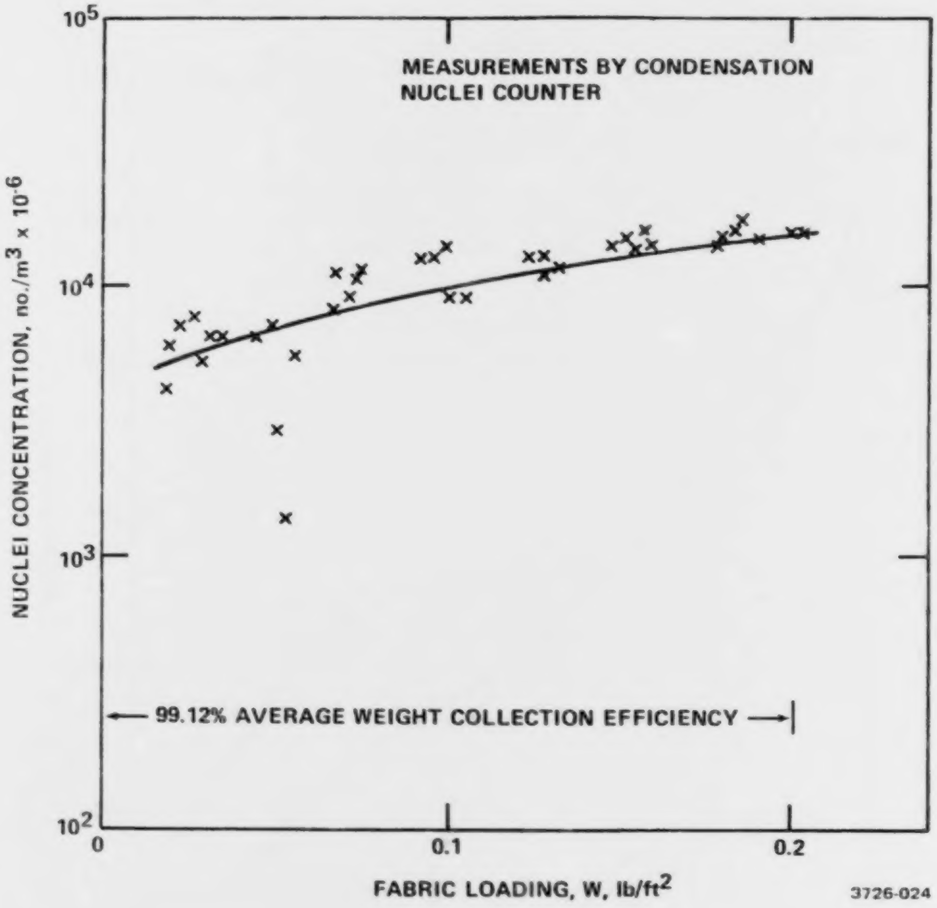


*Figure 8. Effluent concentration vs. filter fabric loading. Sunbury, PN coal-fired boiler fly ash. After Dennis, Cass, and Langley(1975).*

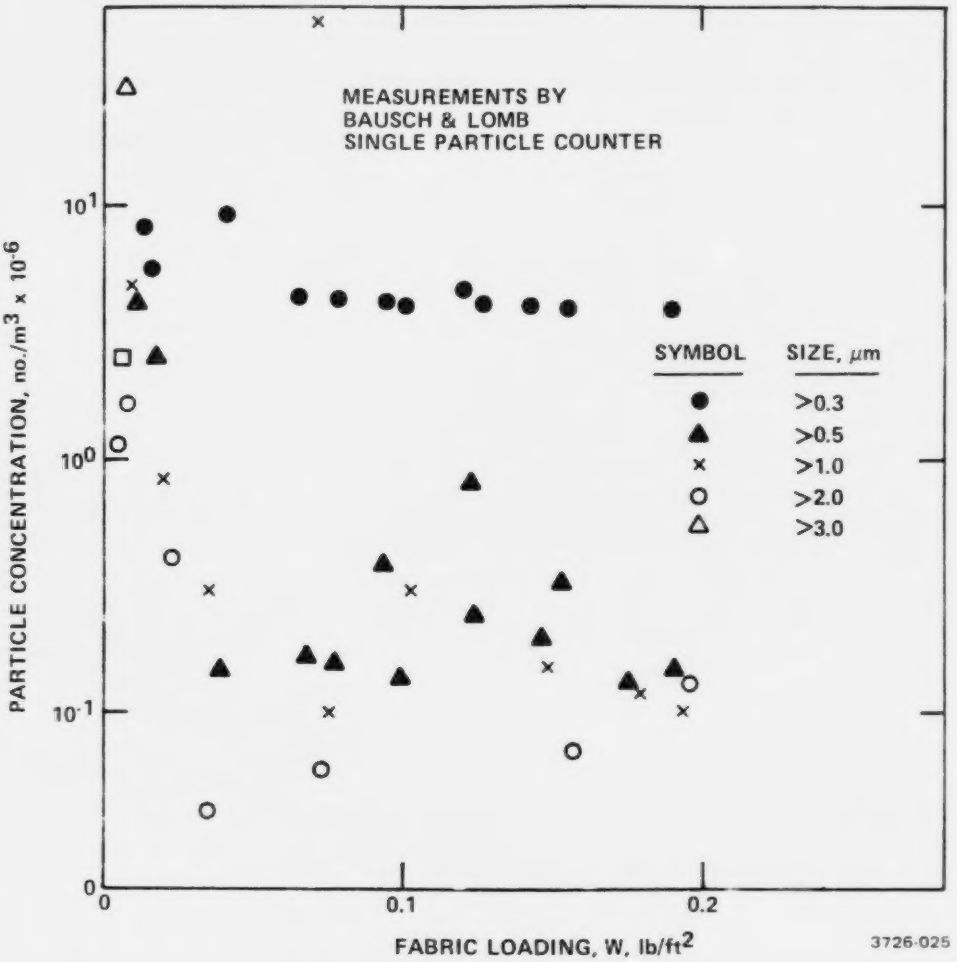


*Figure 9. Effluent concentration vs. filter fabric loading and particle size. Used Nucla, Colorado baghouse fabric and fly ash. After Dennis, Cass, and Langley(1975).*

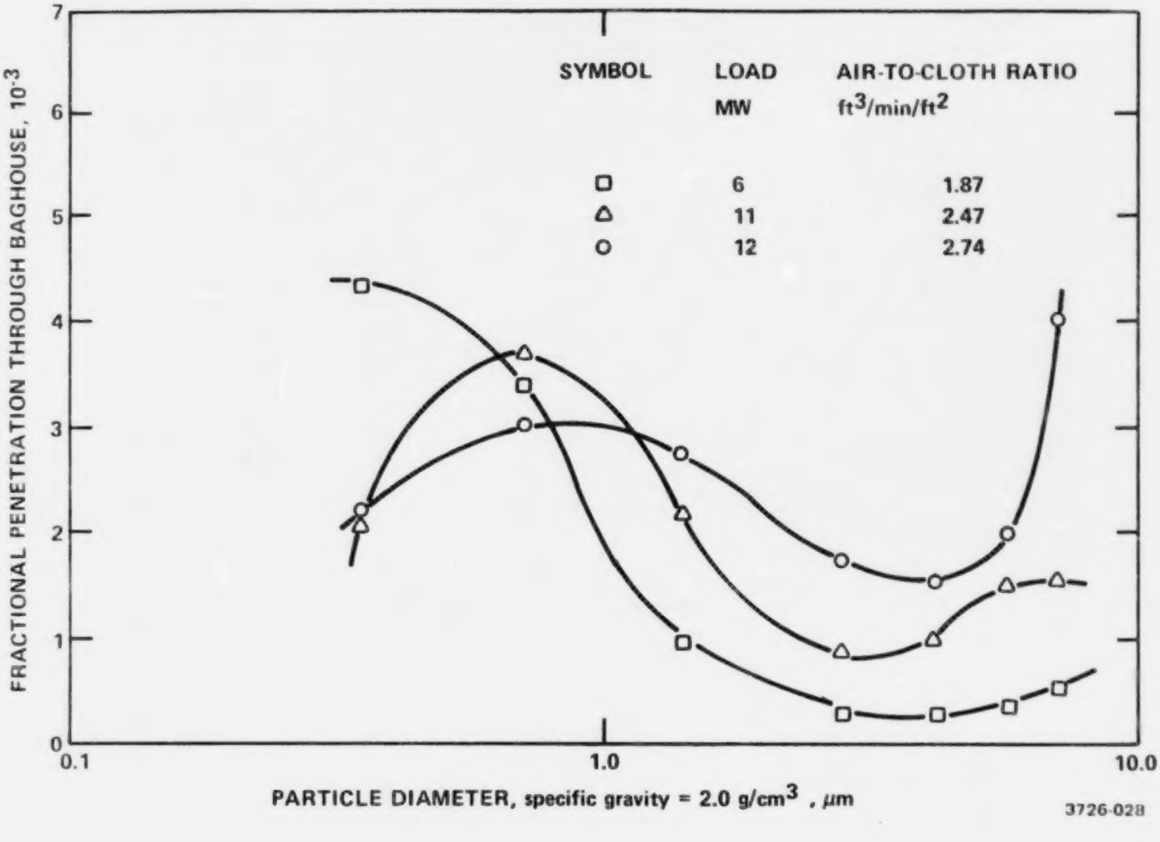


*Figure 10. Penetration as a function of particle diameter and coal-fired boiler load. After Ensor, Hooper, and Scheck(1976).*

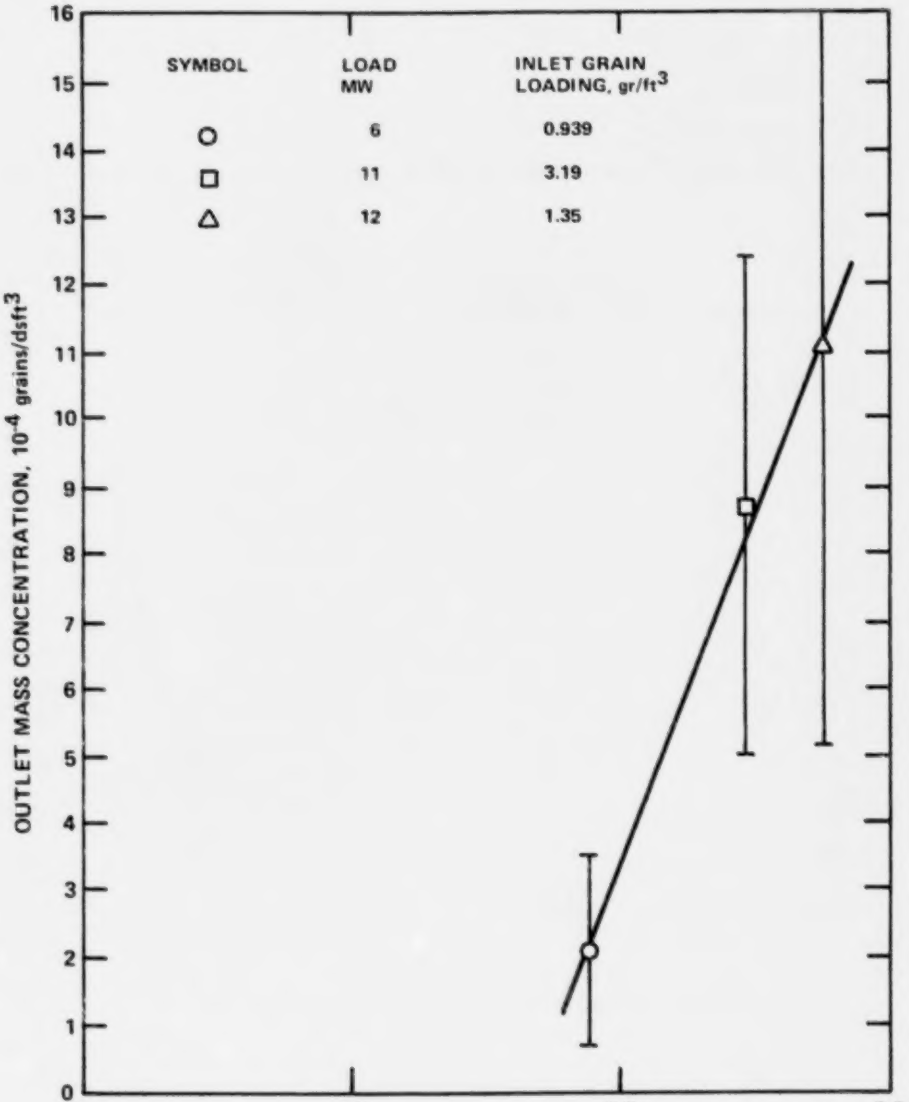
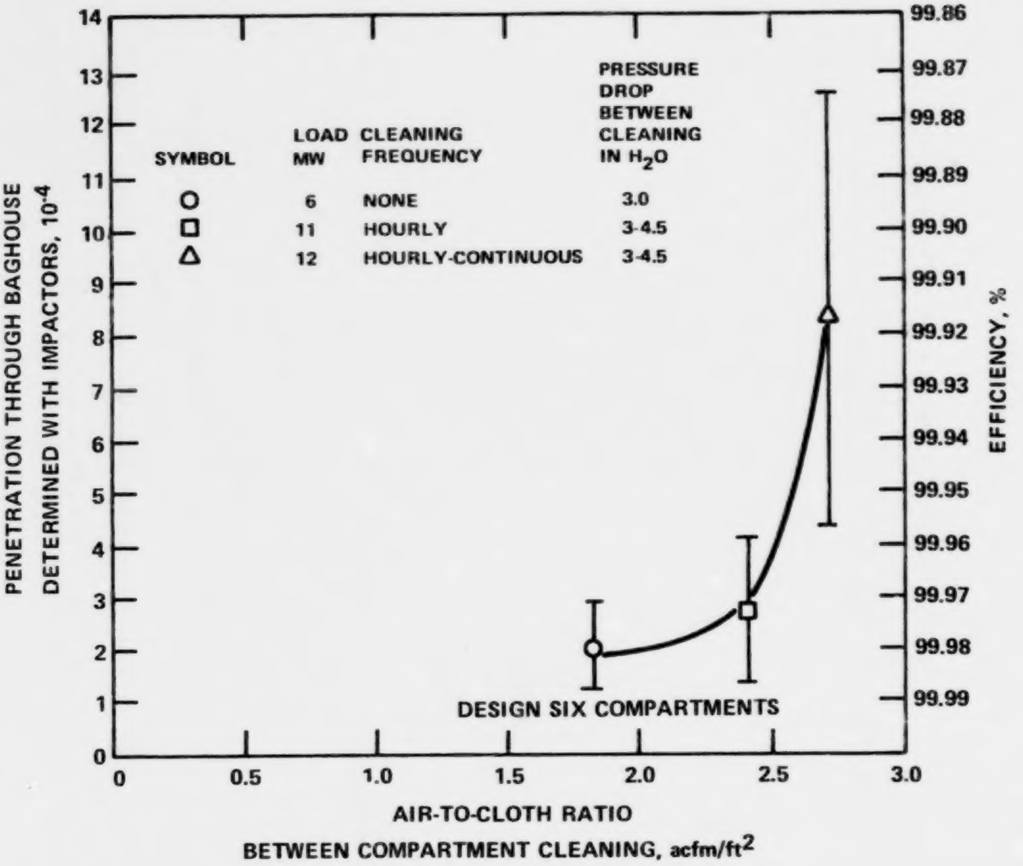


*Figure 11. Average outlet mass concentration (with one standard deviation limits) as a function of air-to-cloth ratio. After Ensor(1976).*



| SYMBOL | LOAD MW | CLEANING FREQUENCY | PRESSURE DROP BETWEEN CLEANING IN $H_2O$ |
|---|---|---|---|
| ○ | 6 | NONE | 3.0 |
| □ | 11 | HOURLY | 3-4.5 |
| △ | 12 | HOURLY-CONTINUOUS | 3-4.5 |

*Figure 12. Penetration (with one standard deviation limits) as a function of air-to-cloth ratio. After Ensor(1976).*

There is some agreement as a result of these findings that emissions from fabric filters are the result of indirect fault processes such as seepage or pinhole leaks, or both, in contrast to failure to collect these particles initially. If this is the case then the following penetration characteristics can be explained (Oglesby, 1977).

a. Greater penetration as a result of higher face velocity can be understood to result from an increased incidence of pinholes at high velocity, as well as from increased pressure drop which may lead to increased seepage.

b. Constant penetration as the dust cake thickens can be understood as a result of the inability of the pinholes to seal after they have been formed. The high air flow rate through these pinholes may tend to keep them open, unless large particles are present.

c. Continuously decreasing penetration is explainable if, as the dust cake builds up, the fabric and dust do not interact to create pinholes.

d. Because these pinholes have no fractionating capability, constant penetration for all particle sizes is explained if particles pass through these holes.

e. Considerable penetration due to seepage may explain the insensitivity of outlet emission rates to inlet concentrations of particles.

### 1.1.3 Filter Fabrics

Two basic types of fabric cloth are used in fabric filters. They are woven cloth and felted cloth.

Woven fabric filters in conventional baghouses usually have air-to-cloth ratios of 1:1 to 5:1. Woven fabric permeability can be varied, thus changing the operating air-to-cloth ratio. Woven fabric permeability, or porosity, is varied by using different yarns, fabric count, cloth weights, and weave patterns. The three basic forms of yarn are monofilament, multifilament, and spun-staple. The basic weaves used for fabric filters are plain, twill, and sateen. See Figures 13 and 14. The more common woven fabric bags are made from cotton, wool, Dacron, Nylon, Orlon, Nomex, polypropylene, Teflon, and fiberglass. Dimensional stability is an important factor in filter fabric. Cotton and wool must be preshrunk and synthetics are usually given a corresponding treatment called "heat-setting." Fiberglass-type fabric bags are treated with silicone, mixtures of silicone and colloidal graphite, or Teflon lubricants to provide protection against flexing of the fabrics and abrasion. These lubricants are effective up to about 290°C.

Felted fabrics serve as filter media and are used in reverse-air and pulse-jet baghouses with air-to-cloth ratios of 6:1 to 16:1. Felted bags are more expensive than woven bags. Wool is the only fiber which will produce a true felt. However, synthetic materials can be needled to function as a felt fiber. Of the materials mentioned above cotton and fiberglass do not yet make felted fabrics.

Fabric filter bags have an average life of 18 to 36 months. Several causes of bag failure include blinding, melting, caking, tearing, burning, decomposition, abrasion, chemical attack, and aging. The circumstances that can lead to bag failure vary widely. Some occur during normal operation; others are due to misapplication or improper operation.

Table I presents a summary of the physical and chemical properties of current leading filter fibers.

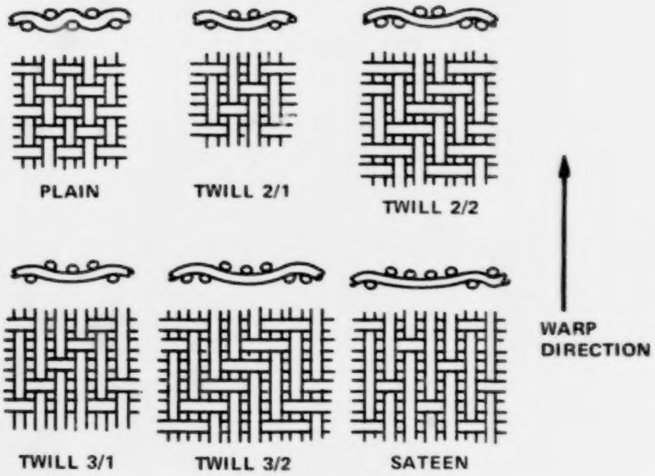
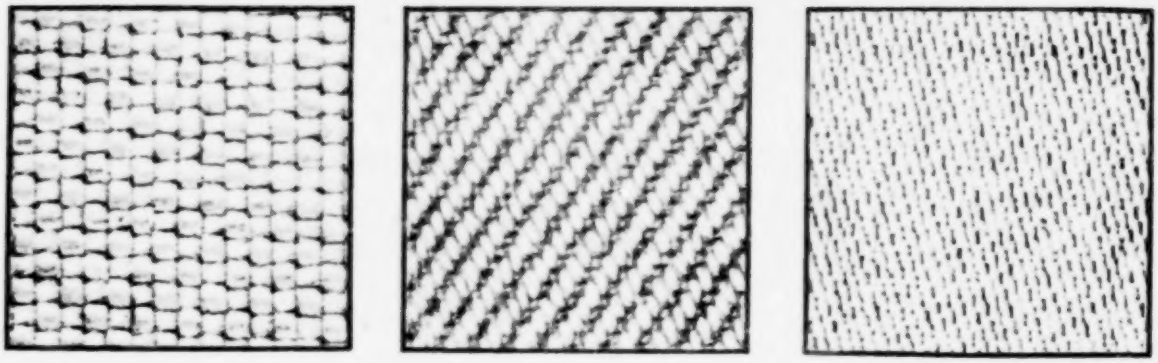

PLAIN
TWILL 2/1
TWILL 2/2
TWILL 3/1
TWILL 3/2
SATEEN
WARP
DIRECTION

*Figure 13. Typical filter cloth weaves.*



PLAIN TWILL SATEEN

*Figure 14. Basic fabric weaves used for woven filter bags.*
*(Courtesy of West Point Pepperell, Industrial Fabrics Division)*

## TABLE I.

## SUMMARY OF PHYSICAL AND CHEMICAL PROPERTIES OF INDUSTRIAL FILTER FIBERS

| | SPECIFIC GRAVITY | MAX RECOMMENDED OPERATING TEMPERATURE °C | RESISTANCE TO ABRASION | RESISTANCE TO MINERAL ACIDS | RESISTANCE TO ORGANIC ACIDS | RESISTANCE TO ALKALIES |
|---|---|---|---|---|---|---|
| Creslan | 1.18 | 135 | G | G | G | F |
| Orlon | 1.14 | 135 | G | G | G | F |
| Nylon | | 93 | E | F | F | E |
| Filtron | | 132 | VG | VG | VG | G |
| Dynel | | 71 | F | VG | VG | VG |
| Nomex Nylon | 1.38 | 232 | E | F | E | G |
| Dacron | 1.38 | 148 | E | G | G | G |
| Polypropylene | .90 | 107 | E | E | E | E |
| Teflon | 2.10 | 260 | F | E | E | E |
| Fiberglass | 2.54 | 288 | P | E | G | G |
| Cotton | 1.50 | 107 | G | P | G | G |
| Wool | 1.32 | 93 | G | F | F | P |

---

E - Excellent
VG - Very Good
G - Good
F - Fair
P - Poor

### 1.1.4 Types of Fabric Filters

Baghouses are characterized according to the method used to remove the particulate matter from the bags. In shaker-type units, the filter bags are hung from a structural framework. The structure is supported so that it will oscillate freely when driven by an electric motor. At set intervals, a damper is used to isolate a baghouse compartment such that there is no gas flow through it. The bags are then shaken for a preset period of time. The collected dust is dislodged from the bags and falls into a hopper from which it is subsequently removed. A shaker-type baghouse is depicted in Figure 3. Figure 15 shows the amount of dust remaining on a bag after several shakes during a cleaning cycle.

Reverse-flow baghouses are equipped with a secondary fan which forces air through the bags in an isolated compartment in the direction opposite to filtraton. This action collapses the bag and fractures the dust layer. When the filter bags are reinflated by being brought back on line, the broken dust layer is dislodged into the hopper. If the main process fan is located downstream of the baghouse, the reduced pressure in the structure may eliminate the need for an auxiliary fan. Sometimes shaking and reverse-flow cleaning mechanisms are combined in the same baghouse unit. This reverse-flow cleaning action is illustrated in Figure 16.

Reverse-pulse fabric filters use a short pulse of compressed air directed from the top to the bottom of each bag. This burst, usually less than 100 milliseconds, aspirates secondary air as it passes through a nozzle or venturi. The resulting air mass expands the bag and expells the collected dust. An example of this cleaning mechanism is diagramed in Figure 17.

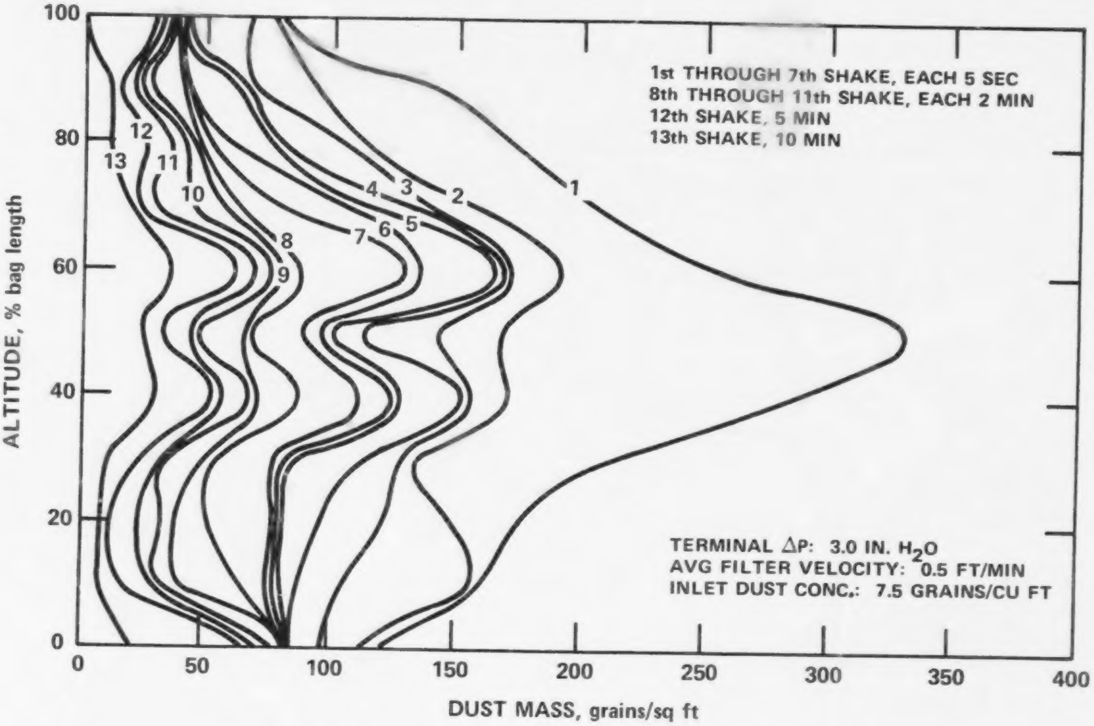


*Figure 15. Residual dust left after several periods of shaking of a fabric filter bag. The dust mass varies along the length of the bag in a manner characteristic of the cleaning technique. After Stephan and Walsh(1960).*

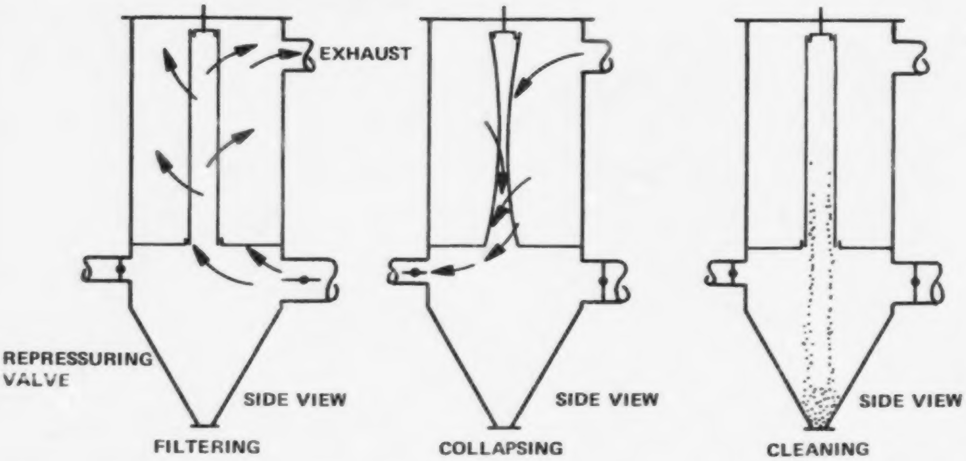

EXHAUST
REPRESSURING VALVE
SIDE VIEW
FILTERING
SIDE VIEW
COLLAPSING
SIDE VIEW
CLEANING

*Figure 16. Reverse air flexing to clean dust collector bags by repressuring.*

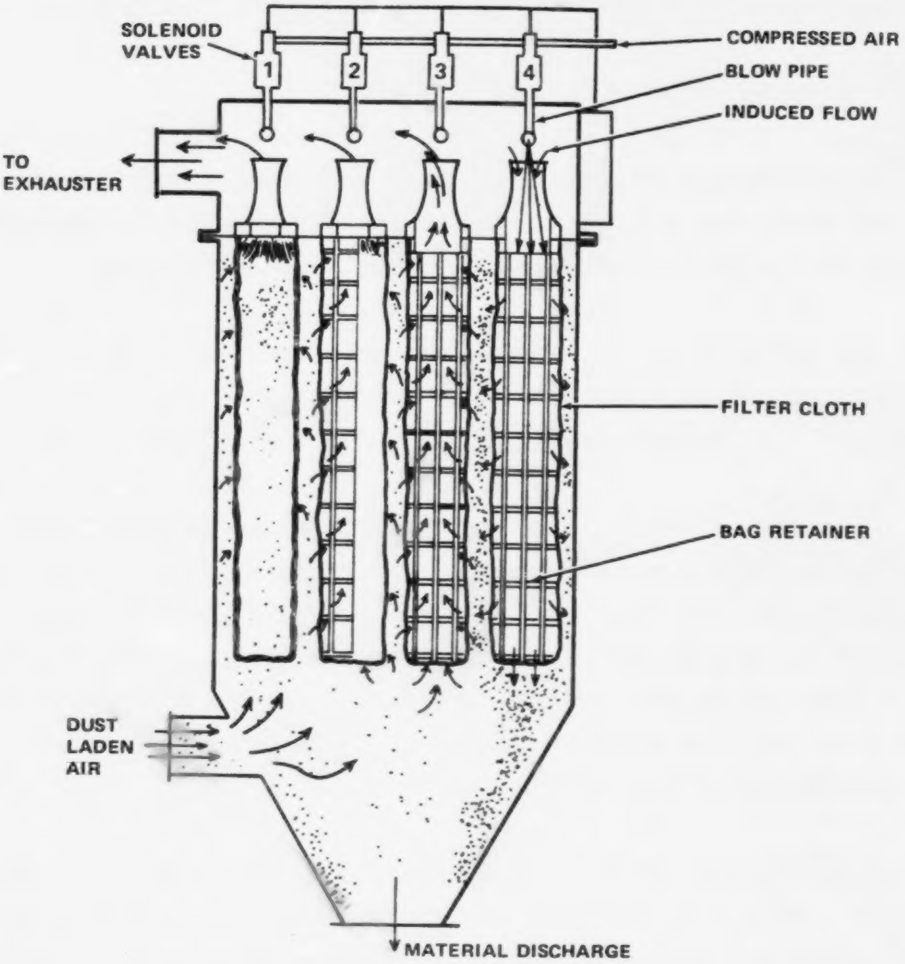

SOLENOID VALVES
1
2
3
4
COMPRESSED AIR
BLOW PIPE
INDUCED FLOW
TO EXHAUSTER
FILTER CLOTH
BAG RETAINER
DUST LADEN AIR
MATERIAL DISCHARGE

*Figure 17. Typical reverse--pulse baghouse during cleaning cycle.*

Shaker and reverse-flow baghouses collect dust on the inside of the bags, while reverse-pulse units collect dust on the outside of the filters. Several hybrid combinations of these three cleaning methods have been developed. They include reverse-flow (shake assist), reverse-flow (vibrator assist), pulse, and pulse (off-line). See Figures 18 and 19.

## 1.2 PARTICULATE SAMPLING FOR FABRIC FILTER EVALUATION

### 1.2.1 General Considerations

Measurements of particle size and concentration are usually made at both the fabric filter inlet and outlet to obtain an accurate characterization of the baghouse performance.

Gas velocities may vary from 15 m/sec to 30 m/sec, in ducts leading toward or away from the baghouse, to as low as 1-5 m/sec in transforms immediately upstream or downstream of the baghouse.

If samples are taken near the baghouse proper, then low gas velocities pose problems because of the difficulty of sampling isokinetically at such locations. Large diameter nozzles are required to sample at low gas velocities when normal sampling train flow rates are used. Conversely, high gas velocities can create situations where nozzles of very small diameter (one or two millimeters) are necessary.

Particulate concentrations at the sampling point may range from 10 $g/m^3$ to 0.001 $g/m^3$ depending upon the type of source and the collection device efficiency. Sampling times to collect accurate data vary approximately inversely with the dust concentration. At baghouse outlets it is not uncommon to sample for twelve hours or longer to collect a sufficient amount of particulate matter. In fact, the particulate concentration can be too low for some instruments to detect or measure. Sampling times can

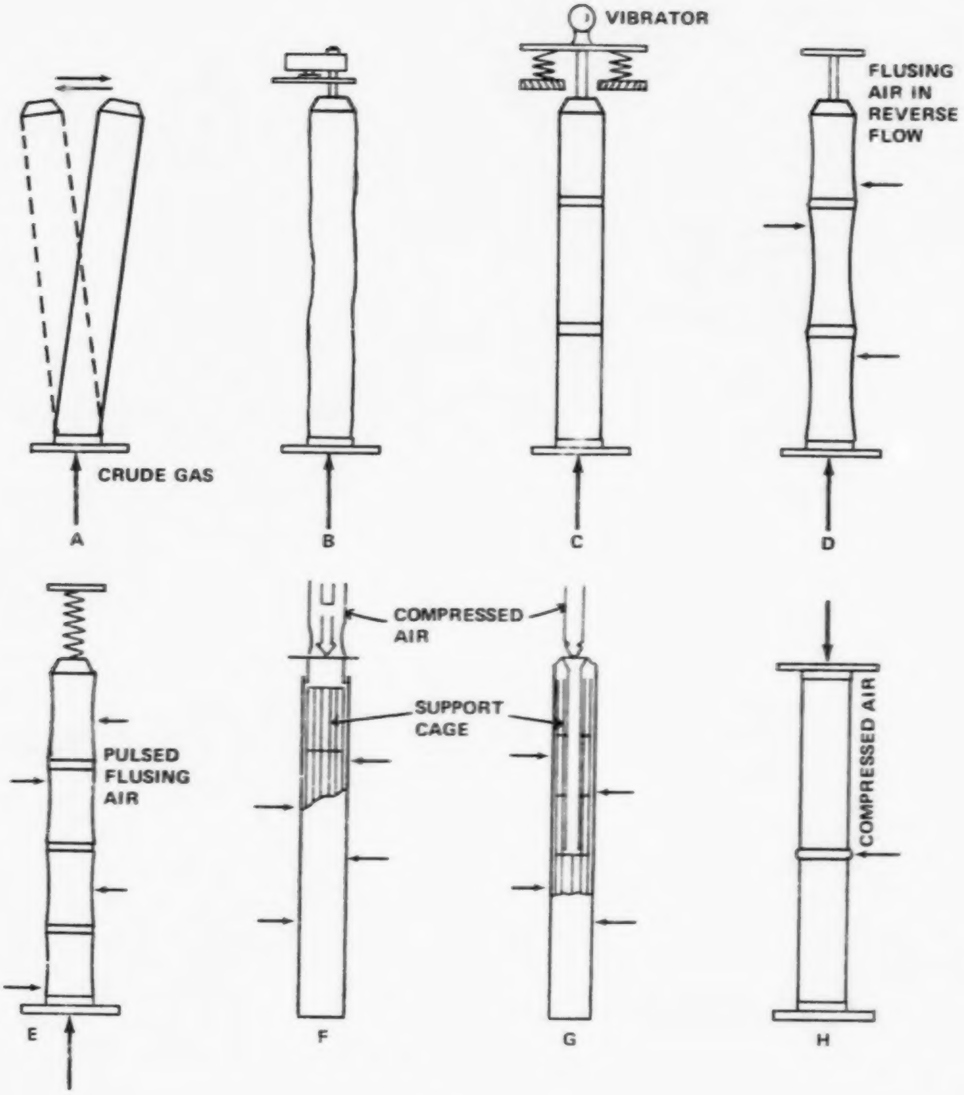

VIBRATOR
FLUSING AIR IN REVERSE FLOW
CRUDE GAS
A
B
C
D
COMPRESSED AIR
PULSED FLUSING AIR
SUPPORT CAGE
COMPRESSED AIR
E
F
G
H

*Figure 18. Cleaning methods for filter bags. (a to c: mechanical cleaning by shaking [rapping mechanism or vibrators], d to h: pneumatic cleaning) After Batel(1973).*

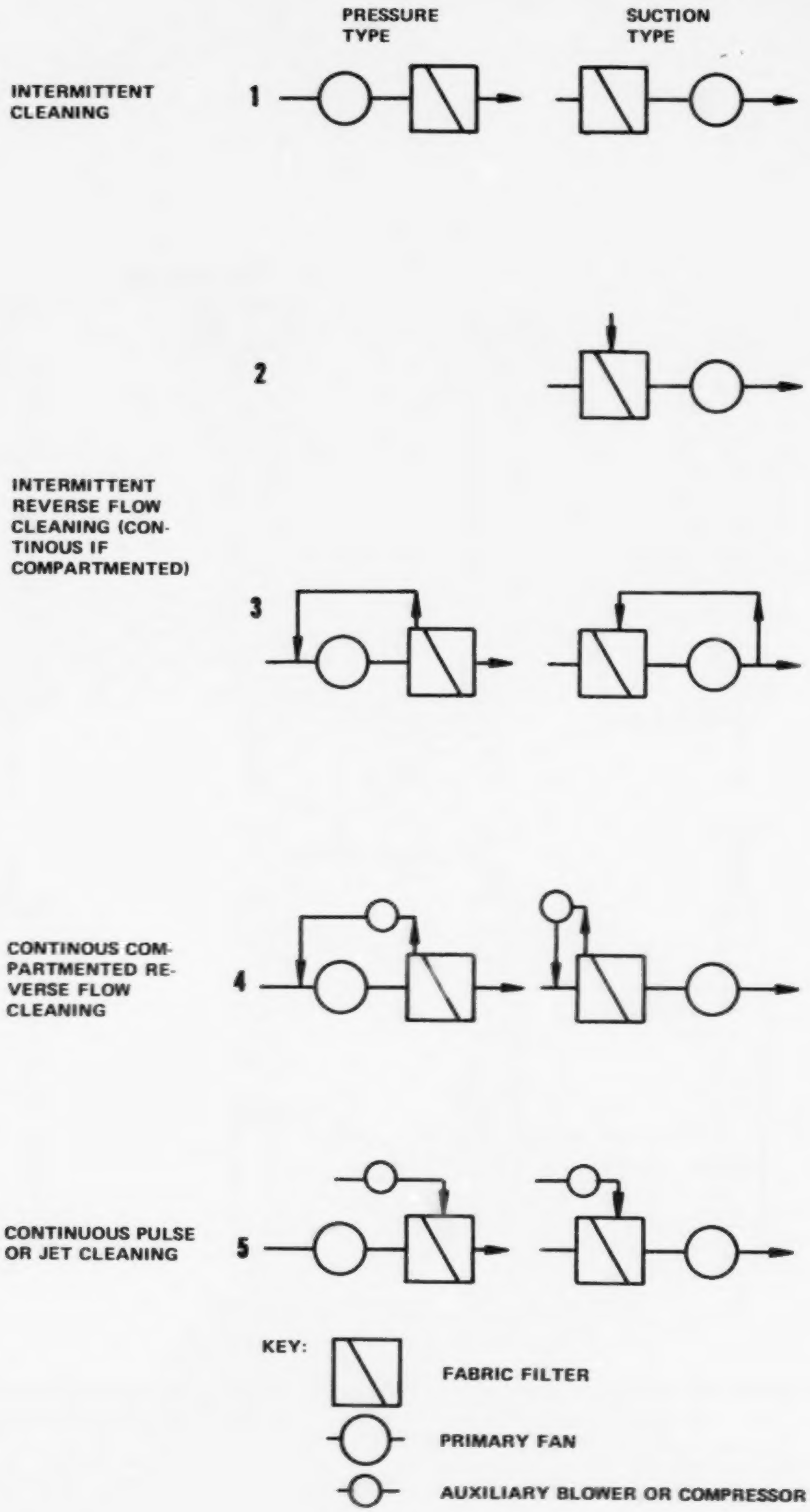

PRESSURE TYPE
SUCTION TYPE
INTERMITTENT CLEANING
1
2
INTERMITTENT REVERSE FLOW CLEANING (CONTINOUS IF COMPARTMENTED)
3
CONTINOUS COMPARTMENTED REVERSE FLOW CLEANING
4
CONTINUOUS PULSE OR JET CLEANING
5
KEY:
FABRIC FILTER
PRIMARY FAN
AUXILIARY BLOWER OR COMPRESSOR

*Figure 19. Types of fabric filter systems depending on cleaning method. After Billings and Wilder (1970).*

also be discontinuous if studies are being conducted to isolate the effects of baghouse compartment cleaning.

At some sources, condensation can occur within the pollution control system or in the stack, and particles may grow larger and change composition, or be created, by this mechanism. These occurrences will cause changes in the observed particle concentration and size distribution.

In many instances the duct dimensions at the sampling location can pose problems, especially if sampling from the top of the duct is required. The duct may be up to seven meters in depth. A hoist must be constructed in some cases to handle probes required to obtain traverse samples near the bottom of such a duct.

### 1.2.2 Particulate Mass Measurements

Measurements of the particulate mass concentration are made by pumping the dust laden gas through a system containing a filter and a means of measuring the volume of the gas sampled. The total material collected on all surfaces within the system is recovered and weighed. This weight, normalized to a unit of gas volume, is the suspended particulate concentration. The samples are collected using a prescribed traversing procedure, which, in effect, yields an approximate integration of the average mass emission rate past a cross-section of the duct or stack. The velocity distribution of the gas is also measured as part of the test. The gas flow rate and velocity distribution and the particulate mass concentration are used to calculate the mass flow rate or emission rate at the point of interest. Measurements of the mass flow rate are made at both the inlet and outlet to determine the fabric filter's collection efficiency. Section 2.2 contains a summary of the methods for conducting mass measurements. Appendix B describes in detail the EPA Method 5 particulate mass measurement procedures.

### 1.2.3 Particle Sizing Techniques

Measurements of particle size distributions in industrial flue gas streams are made for several reasons. The aerosol must be characterized as completely as possible in order to assess the potential for adverse health or environmental effects; emission measurements can be useful as a process monitor; and the aerosol particle size distribution must be known in order to completely quantify the behavior of a control device. Also, particle size measurements on uncontrolled sources are sometimes useful in fabric filter design.

In recent years the emphasis in pollution control has been placed on "fine" particles which are defined by the EPA as those particles having aerodynamic diameters smaller than three micrometers. Fine particles are more difficult to control than large particles, and because they are respirable, may constitute a greater hazard to health.

Several techniques must be applied if information on particle size over a wide range of diameters is required, or if real time data is desired. As a general rule, most particle sizing techniques yield accurate information over approximately a factor of ten range in particle diameter. The particle size range with which this manual is concerned is 0.01 to 10 micrometers diameter. Therefore, several techniques must be employed.

Section 2.3 contains a summary of the instruments that are available for particle sizing, as well as a discussion of their applicability to specific tests. Appendices C and D contain detailed descriptions of the procedures for measurement of particle size distributions in flue gases.

# 2. TECHNICAL DISCUSSION

## 2.1 MECHANICAL CHARACTERIZATION OF A FABRIC FILTER INSTALLATION

In order to perform a comprehensive analysis of the performance of a baghouse it is necessary to obtain information on the design and operating parameters of the complete installation. These data are generally used, with the test results, to assure compliance with regulations, or to make comparisons with other fabric filter evaluations or with performance predictions of theoretical or empirical models. Plant or process operating data should also be obtained as part of each test program, and correlated with the control device performance. Plant data are routinely recorded by the plant personnel and arrangements can usually be made to obtain copies of these records or to have a member of the test crew record the data during a test.

In the remainder of Section 2.1, the nomenclature that is used to described fabric filters is defined and a number of parameters that should ideally be measured or noted during a baghouse evaluation are listed. Some of the data listed are essential to a meaningful evaluation, while some may be difficult or impractical to obtain and must be sacrificed. Also, some installations may have individual peculiarities requiring that additional data be taken. Although this manual is intended as a comprehensive guide, there is no substitute for intelligent and experienced judgement in the discrimination of essential from nonessential data.

### 2.1.1 Mechanical Design and Operating Data

The objective of a baghouse design is to combine the component parts into an effective arrangement that results in an optimum collection efficiency at the least operating expense. Experience, efficiency requirements, and economics generally dictate the best arrangement. Fabric filter technology can be improved through the study and comparison of many of the items listed below and in the following sections.

For completeness, determinations of the following items pertaining to the physical layout and mechanical operation of the baghouse should be made during the test. This data may be obtained by observation, from plant personnel, or from manufacturers' literature.

Number of baghouses -- A baghouse is a metal box-like structure with inlet and outlet gas manifolds and which contains the filtering elements.

Dimensions of the baghouse -- The physical height, length, and width of each baghouse should be noted.

Inlet and outlet manifolds -- The physical dimensions of the gas inlet and outlet manifolds should be noted; whether they are round or rectangular; whether they have any bends that might cause poor particulate concentration distribution; whether there are any flow distribution devices or baffles in the ducts leading to the baghouse proper. Any or all of these items can affect the observed inlet and outlet dust concentration measurements. Sampling ports are usually located in these inlet and outlet ducts. If this is the case, their size, shape, number, and location should be noted.

Number of baghouse compartments -- Each large, nonreverse pulse cleaned baghouse is divided into several independent compartments. During a complete cleaning cycle, each compartment is taken off line in turn and the bags are cleaned. When one compartment is returned on line, another compartment is taken off line for cleaning.

Total gas volume, temperature, and moisture content -- The average total gas volume through the baghouse, its temperature, and normal moisture content can usually be obtained from plant data. During a normal evaluation procedure, however, these data are obtained as part of the mass concentration (EPA Method 5) measurements.

System air movers -- It is usually worth noting the amount of fan capacity and also whether the system is induced draft or forced draft. The amount of air flow through the baghouse will be affected by the buildup of dust on the bags and their cleaning cycle. The fan capacity should be capable of handling the maximum filter pressure drop anticipated during normal operation.

Compartment access -- The accessibility to the baghouse compartments should be determined. This will indicate how readily old bags can be replaced with a minimum of down time.

### 2.1.2 The Fabric Filter Bags

The entire baghouse is designed around the filtering element or bag; thus, detailed information on their design, structure, and application is required.

Total number of bags -- The total number of bags in each baghouse should be noted.

Number of bags per compartment -- The number of bags in each baghouse compartment should be noted.

Bag shape and size -- The bag shape as well as the length and diameter should be determined.

Bag construction -- Several items with respect to the construction of the bags should be noted. These include seams, closures, seals, clamps, cuffs, reinforcements (cage, rings, or frame), attachments and thimbles. This information can usually be obtained from plant personnel or manufacturers' literature.

Bag placement and spacing -- The arrangement of the bags in each compartment should be determined as well as their spacing, bag to bag and bag to wall. These parameters affect bag wear, air movement, and ease of bag replacement.

Type of bag cleaning mechanism -- The type of mechanical action by which the dust is removed from the bags should be noted. Currently available types include (1) off-line, gravity; (2) off-line, collapse; (3) off-line, collapse, reverse-air; (4) off-line, shake or vibratory; (5) reverse-air; (6) pulse-jet; and (7) reverse-air, shake assist.

Cycle for cleaning bags -- The sequence of compartment bag cleaning should be noted, including how often each compartment is taken off-line for cleaning, the duration of each cleaning cycle, and the amount of each cycle used for shaking, reverse air, pulse, etc.

Pressure drop to initiate cleaning -- Generally bag cleaning commences when the pressure drop across a compartment reaches a predetermined value. Once this pressure drop is attained, the compartment cleaning cycle commences automatically, and continues until the pressure drop either has been lowered to a preset level or a preset length of time has elapsed. Generally each manufacturer has a particular type of cleaning system developed that is somewhat unique from any other.

### 2.1.3 Filter Fabrics

The filtering element in the baghouse installation is the fabric. A wide variety of commercial synthetic and natural fibers have been developed to cope with many different types of industrial situations.

Fiber material -- The type of industrial process will generally determine the fiber material that is used in the fabric filters. Many synthetic materials have recently been marketed which will withstand temperatures above 100°C. A list of the more commonly used fibers can be found in Section 1.1.2.

Fiber and fabric characteristics -- There are many properties of the filter fabric that can be specified; however, they are not all essential to a complete evaluation. Some of the information also may be very difficult to find, except through the manufacturer's literature. For completeness, a list of these properties is presented below:

Surface properties
Fiber size, length/thickness, texture
Specific gravity
Strain, recovery, creep
Flexibility, elasticity
Stiffness
Hardness, deformation
Modulus, compliance
Moisture regain
Friction, cohesiveness
Electrical properties, dielectric constant
Surface asperities
Physical resistance to the environment
Chemical resistance to the environment
Age effects, degradation

Bacteriological effects
Polymer additives, treatments
Yarn twist, lay
Staple, filament configuration
Number of fibers/yarn
Thread used to sew
Weave style
Crimp, geometric properties
Porosity
Yarn count, warp, fill
Yarn tension
Interyarn spacing
Available free fiber

Fabric treatments -- To aid in a longer bag life most commercial filter bags are treated with special lubricants, such as graphite and Teflon, to cut down on fiber to fiber and bag to bag abrasion. Also most commercial fibers are treated or "heat-set" to reduce stretching or shrinking at elevated temperatures.

Fabric thickness and weight -- The weight of the fabric should be noted. It is measured commonly in ounces per square yard. This will give an indication of the durability of the product.

Air-to-cloth ratio or air flow permeability -- The air-to-cloth ratio gives the volume of air per minute passing through one square foot of cloth at a predetermined pressure drop. This is also the filtering velocity. A knowledge of this quantity is important in determining the final system operating pressure drop, as well as the pressure drop at which cleaning is initiated. Generally the pressure drop across each baghouse compartment is monitored. Figures 10 and 11 show experimental data indicating the relationship between air-to-cloth ratio and fabric filtering efficiency at a coal-fired boiler.

### 2.1.4 Dust Removal Systems

It is important during any fabric filter evaluation to determine that the dust removal system is working properly and to specifications. If the system is not working properly, discussions should take place with plant personnel to see whether these problems can be fixed in a reasonable time span. If this is not the case, then testing should be delayed.

Hoppers are used to collect and store dry particulate which is removed from the bags. If the hoppers are allowed to overflow, the collected dust may reach the level of the bags and cause blinding, chafing, or chemical attack.

Several types of systems exist for removal of dusts accumulated in hoppers. These include container removal, dry vacuum, wet vacuum, screw conveyors, and scrape bottom systems.

As much information about the dust removal system should be reported as is practical.

### 2.1.5 Baghouse Operation-General Maintenance Considerations

Although noting general maintenance procedures is not necessarily a part of an evaluation procedure, the status of baghouse operation at the time of testing will depend on the previous operation and maintenance schedule. Thus a brief description of these procedures is in order.

One way to minimize operating and maintenance costs while prolonging the life of the fabric filters is to keep the air flow and temperature into the filters as low as possible, limited only by the danger of reaching the dewpoint. The day-to-day use of a filter system requires frequent observation and occasional adjustments in order to determine and adhere to the best overall compromise between performance, bag life, power, costs, etc.

Use of instruments -- A single manometer across each baghouse compartment generally provides all the data necessary on the operation of that section of the unit. It indicates the permeability of the cloth, how heavy the dust deposit is before cleaning, how complete the cleaning is, and whether the fabric is starting to plug or blind. It indicates what surges in velocity the dust deposit is under going and whether there is any flow through the cloth during the cleaning cycle. It is advisable to have a list of normal differential pressures through one filtration cycle, as a means to quick detection of later trouble. A high differential pressure may mean:

- an increase of air flow
- the beginning of blinding of the cloth
- hoppers so full as to block off the bags
- condensation in the cloth
- cleaning mechanism inoperative

while a low pressure differential may mean

- the fan is slowing down, perhaps due to belt slippage or fan motor problems
- broken or unclamped bags
- plugged inlet ducting or valves closed
- leakage between sections of the filterhouse.

Other monitoring instruments can be nearly as valuable, since transients frequently occur in pressure, flow rate, temperature, humidity.

Flow variations -- At processes requiring multiple or variable dust pick-up points, there will be variations in flow rate and filtering velocity. All the branches of the inlet ducting may be open or some may be shut off, depending on plant activity. Also, while one collector is down for its cleaning cycle, another may be down for bag changes or still another for inspection. These system changes affect the flow through each filter. Too much flow through too few bags amounts to an overload or too high a filter velocity, leading to insufficient filtering, plugging of the cloth, loosening or bursting of the bags. Too low a flow is a frequent cause of condensation.

Cleaning cycle -- As the cloth ages, adjustments in the cleaning cycle either in the amount or intensity of cleaning or in the length of the cleaning cycle are generally necessary. To prolong the life of the fabric, as little cleaning as necessary is advisable; but enough cleaning is required to keep the differential pressure at an economic level. There is a point of optimization, although this may be difficult to locate in practice. Operating at the point of minimum cleaning is indicated by a gradual build-up of differential pressure, perhaps over a period of a few days. Before the pressure gets too high, a slight increase in cleaning action should reverse the trend in pressure. After a few cycles or a few hours the pressure should reach a sufficiently low level to reduce the cleaning action. If this gradual fluctuation in differential pressure is not observed, it may mean that the fabric is being overcleaned. Changes in fabric condition or process will undoubtedly cause the cleaning requirements to shift from week to week.

Types and frequency of fabric filter problems -- A survey in the late 1960's obtained information on specific problems encountered with fabric filter installations. Table II lists 23 types of operational problems encountered by the installations surveyed (Billings and Wilder, 1970). They have been grouped into five causality categories as follows:

| | | | | | |
|---|---|---|---|---|---|
| 1. | Fabric-dust interactions | 9 | types of problems | 60 | problems reported |
| 2. | Filter element difficulties | 2 | " | 6 | " |
| 3. | Filter element-hardware interactions | 5 | " | 12 | " |
| 4. | Collector design problems | 5 | " | 27 | " |
| 5. | System design problems | 2 | " | 7 | " |
| | | 23 | types of problems | 112 | problems reported |

## TABLE II

## TYPE AND FREQUENCY OF FABRIC FILTER PROBLEMS REPORTED

| | | Frequency |
|---|---|---|
| 1. | Fabric - dust deposit interactions | |
| | a. interstitial deposit related abrasion, wear | 8 |
| | b. flexture wear failure | 10 |
| | c. seeping | 4 |
| | d. blinding | 14 |
| | e. burning, heat | 6 |
| | f. holes, pinholes, shot holes | 6 |
| | g. hydroscopicity | 4 |
| | h. condensation | 5 |
| | i. deposited dust hardens, cake tears, cracks bag | 3 |
| | Subtotal | 60 |
| 2. | Fabrication failures not particularly related to dust interaction, mechanical | |
| | a. seams, sewing | 2 |
| | b. tears at top | 4 |
| | Subtotal | 6 |
| 3. | Design or maintenance failures related to tensioning, supports, rings, collars, or cleaning device interactions | |
| | a. chafe on housing or other bags | 3 |
| | b. tensioning, bags too loose | 1 |
| | c. cage, wire, ring abrasion, wear (also dust related), support mechanism interact | 5 |
| | d. cleaning carriage bag wear | 1 |
| | e. seals around cloth-metal collars | 2 |
| | Subtotal | 12 |

TABLE II (Continued)

| | | |
|---|---|---|
| 4. | Collector design problems, internal, external mechanisms, incl. auxiliaries, (ex. pipes, hoods) | |
| a. | unable to enter collector to service or maintain during operation | 4 |
| b. | hole detection problems or performance effluent monitor | 3 |
| c. | hopper dust sticking, holdup, screw conveying plug | 6 |
| d. | internal mechanism wear | 4 |
| e. | external mechanism wear, timer, shaker, fan, bearings, doors, seals, wall failure | 10 |
| | Subtotal | 27 |
| 5. | Dust collecting system design problems, external to collector (incl. pipes, hoods) | |
| a. | piping, elbows, abrasion | 4 |
| b. | hood inlet control poor | 3 |
| | Subtotal | 7 |
| | Total: | 112 |

## 2.2 MASS EMISSION MEASUREMENTS

### 2.2.1 General Discussion

As part of most fabric filter evaluation studies, the particulate collection efficiency is determined experimentally by making measurements of the mass concentration and gas flow rate at the baghouse inlet and outlet.

A number of test procedures have been developed for performing mass emission measurements on process streams. All of the "standard" sampling trains are similar and basically are composed of a nozzle, a probe, a filter, one or more devices for monitoring gas flow, and a pump. Generally pitot tubes and thermocouple assemblies are also used to measure the gas velocity and temperature. The nozzle is streamlined to minimize flow disturbances and the diameter is chosen for isokinetic sampling. The probe must be rigid so that the nozzle can be positioned accurately at the selected sampling points.

Filters of various compositions and geometries are used, although glass fiber is most common. Any filter chosen must be an efficient collector of submicron particles. A gas meter and sometimes a calibrated orifice are used to measure the gas flow rate and total volume sampled. Condensers are used to measure the water vapor content, as well as for pump and meter protection. In some instances impinger bubblers and liquid traps are used and the contents analyzed after sampling for various volatile elements.

Many emissions contain substances which condense at temperatures well above ambient to form solid or liquid particles. Care must be taken that the temperature in all parts of the system upstream from the filter be kept at temperatures high enough to prevent condensation. Also, there is sometimes considerable depo-

**Preceding page blank**

sition on the surfaces of the nozzles, probes, etc., and these must be thoroughly cleaned as part of normal sampling and analysis.

Three systems which are commonly used to measure mass emissions are described in the following paragraphs.

### 2.2.2 EPA-Type Particulate Sampling Train (Method 5)

Official performance testing of control devices on stationary sources in the United States must be conducted with the "EPA Method 5 Sampling Train" (Federal Register, 1977) illustrated in Figure 21. A heated sample probe is used to transport the particulate sample to a glass fiber filter which is maintained at 120 ± 14°C. A reverse type pitot tube is attached to the probe to insure that isokinetic sampling conditions are maintained during a traverse of the duct or stack. According to the EPA method the glass fiber filter must have a penetration value equal to or less than that of MSA 1106 BH (approximately 0.05% for a standard 0.3 μm DOP aerosol penetration test at 5 cm/sec face velocity). Gases, vapors, and any particles that penetrate the heated filter enter a series of impingers or condensers that are immersed in an ice bath. They trap the uncombined water that is present in the gas stream so that the moisture content can be determined; they also prevent the hot, humid gases from entering the gas metering system and pump.

After the traverse, the filters are dried and the probe is washed to remove and collect particulate matter from the probe walls for subsequent analysis. The EPA Method 5 requires that acetone be used for the probe wash. This creates a problem when the long metal probes are hot from sampling, so distilled water, with the Administrator's approval, is often used instead of acetone. The probe wash liquid is collected and evaporated to dryness so that the amount of particulate matter removed from the probe can be weighed. This weight, and the weight of the parti-

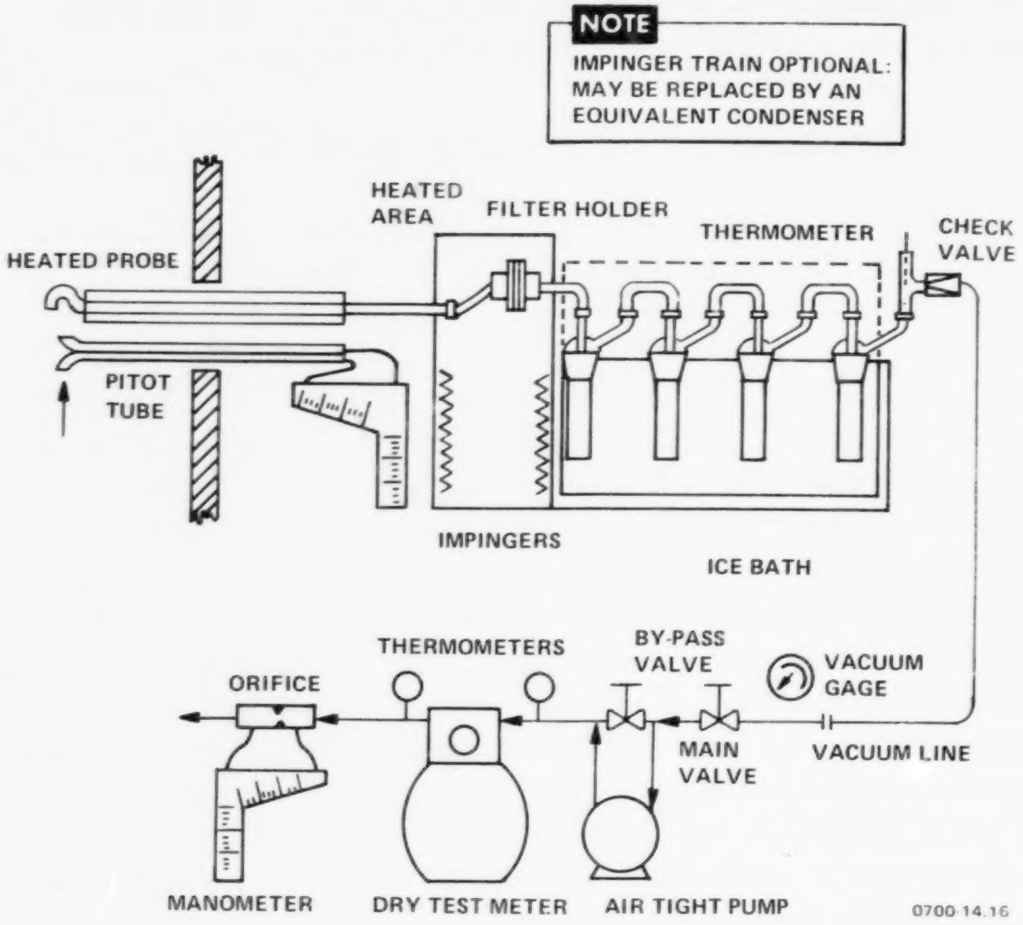

NOTE
IMPINGER TRAIN OPTIONAL: MAY BE REPLACED BY AN EQUIVALENT CONDENSER
HEATED AREA
FILTER HOLDER
THERMOMETER
CHECK VALVE
HEATED PROBE
PITOT TUBE
IMPINGERS
ICE BATH
THERMOMETERS
BY-PASS VALVE
VACUUM GAGE
ORIFICE
MAIN VALVE
VACUUM LINE
MANOMETER
DRY TEST METER
AIR TIGHT PUMP
0700-14.16

*Figure 20. EPA Method 5 Particulate Sampling Train.*

culate matter on the filter, and the measured gas flow are used to determine the mass emission rate of the source. Approved systems of this type are currently available in low (230-472 $cm^3$/sec) (0.5-1 $ft^3$/min) and high (up to 170 LPS) (6 $ft^3$/min) flow rate configurations. A modification to the EPA particulate sampling procedure (Method 17) has been promulgated to allow the use of in-stack filters when testing Kraft pulp mills (see Appendix E).

### 2.2.3 ASTM-Type Particulate Sampling Train

The American Society of Testing and Materials (ASTM, 1977) has described a particulate sampling train which is illustrated in Figure 22. The main difference between this method and the EPA Method 5 is the use of an in-stack particulate filter. With this arrangement, a thimble-shaped filter is used to sample high mass concentrations, and a conventional, disk-shaped, filter is used for low mass concentrations. It is important to heat the filter holder to insure that the filter temperature is maintained above the dew point temperature if condensible vapors are present in the gas stream. The advantage of this system is that the particles are trapped before they enter the probe and a probe wash is not required. Also, external heating of the filter is often unnecessary. A condenser and gas cooler are still required between the probe and the gas metering system. The pitot tube, pump, and other parts of the system are similar to the EPA Method 5 Sampling Train. The thimble-filter system is often used in engineering tests to evaluate the performance of a control device.

### 2.2.4 ASME-Type Particulate Sampling Train

The American Society of Mechanical Engineers (ASME, 1957) has described in its Power Test Codes the use of a sampling train to measure particulate emissions from industrial sources. To meet the ASME specifications the particulate sampling train must have the following parts:

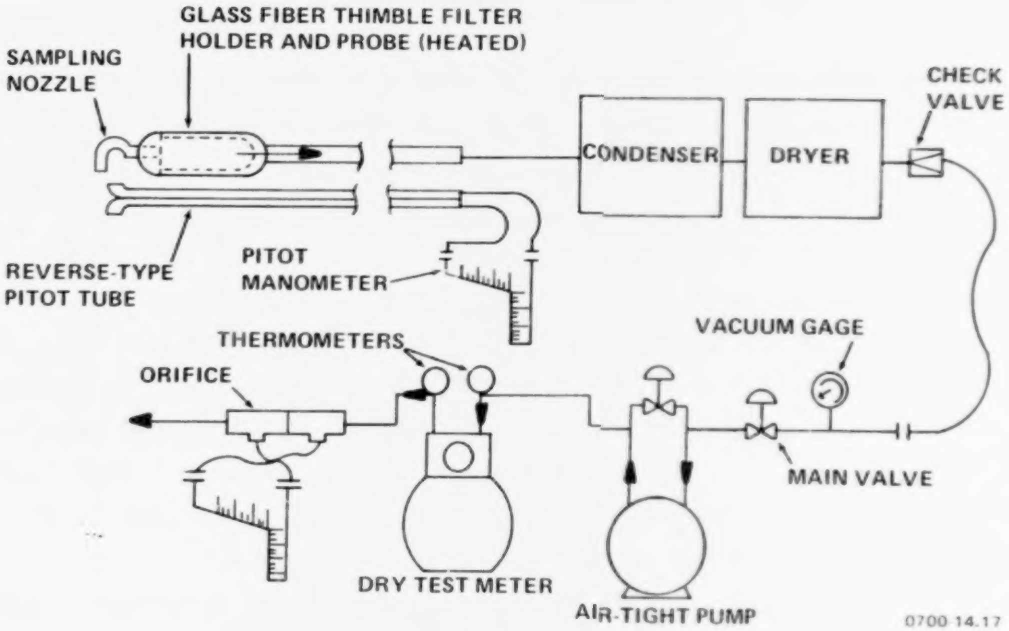

GLASS FIBER THIMBLE FILTER HOLDER AND PROBE (HEATED)
SAMPLING NOZZLE
CHECK VALVE
CONDENSER
DRYER
REVERSE-TYPE PITOT TUBE
PITOT MANOMETER
VACUUM GAGE
THERMOMETERS
ORIFICE
MAIN VALVE
DRY TEST METER
AIR-TIGHT PUMP
0700-14.17

*Figure 21. ASTM Type Particulate Sampling Train.*

A tube or nozzle for insertion into the gas stream and through which the sample is drawn.

A filter (thimble, flat disk, or bag type) for removing the particulate. For the purpose of the Power Test Code, 99.0% efficiency by weight is satisfactory.

A means of checking the equality of the velocity of the gas entering the nozzle and the velocity of the gas in the flue at the point of sampling.

A method by which the quantity of gas sampled is determined.

A pump for drawing the gas stream through the nozzle, filter, and metering device along with the necessary tubing. It is important that the temperature of the gas be above the dew point until after it has passed the filter.

This sampling procedure is not very restrictive. Both the EPA and ASTM particulate trains comply with the ASME requirements.

### 2.2.5 General Sampling Procedures

Because the EPA Method 5 is required for most particulate compliance testing, it is the only one discussed in this section and in Appendix B, where the details of performing a mass emissions test are described.

The general sampling procedure outlined herein is presented in the EPA Test Method 5 "Determination of Particulate Emissions from Stationary Sources." (See Appendix E.) Before sampling, however, it is necessary to determine the number of sampling points appropriate for the particular duct or stack under consideration. EPA Test Method 1 (Federal Register, 1977) "Sample and Velocity Traverses for Stationary Sources" (see Appendix E) describes the computations to determine the number of sampling points for both the velocity traverse and mass sampling traverse. The number of points will depend on the size and shape of the duct. If the velocity traverse indicates that the velocity profile in the duct is very unstable, the number of sample points should be increased to obtain a more accurate integrated mass emission rate (see Figure 23).

The use of the S-Type pitot tube, and its calibration for measuring the stack gas velocity and flow rate, is described in the EPA Test Method 2 (Federal Register, 1977) "Determination of Stack Gas Velocity and Volumetric Flow Rate (Type S Pitot Tube)." (See Appendix E.) The S-Type pitot tube is used because it is less susceptible to clogging in high dust loading environments. It is also advantageous to perform a temperature traverse of the duct during the velocity traverse. This can be easily accomplished by attaching a thermocouple temperature sensor near the end of the pitot. During actual mass sampling both the temperature and gas velocity are monitored to allow isokinetic sampling at each traverse point.

Mass sampling at fabric filter installations can pose problems because of the large difference in dust concentration between the inlet and outlet ports. Baghouse inlet mass loadings are usually high, while outlet loadings can be up to a factor of $10^3$ smaller. Extended sampling times are required in many situations at baghouse outlets. Problems in obtaining average inlet and outlet mass loadings can also occur at sources which have cyclical operations. However, the outlet mass concentrations at several baghouses remain fairly stable even if the inlet concentration is fluctuating. The person preparing for a sampling procedure must take all of these variables into account when designing a test plan.

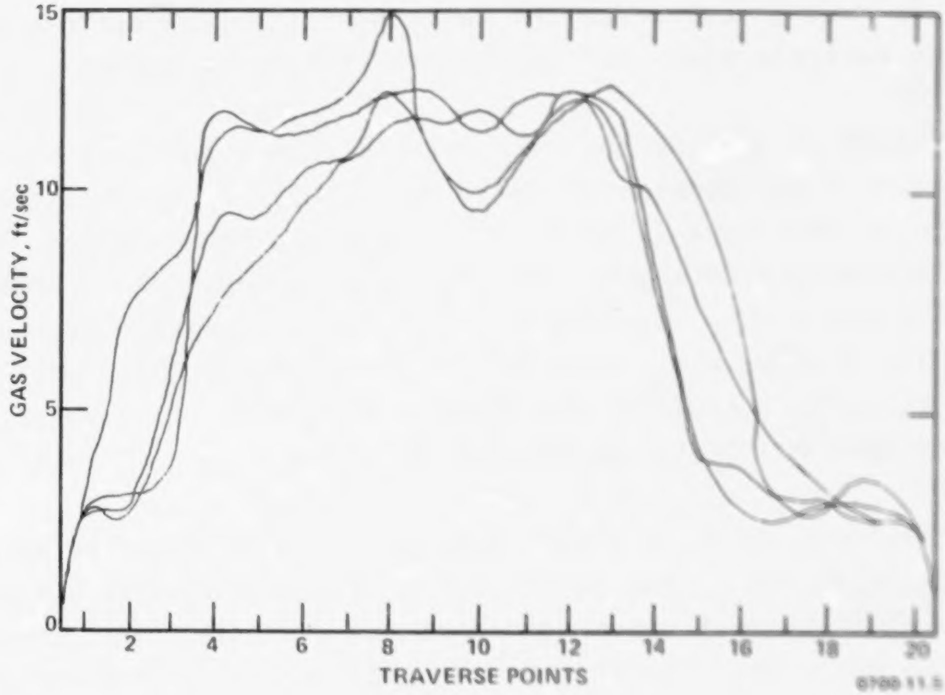


*Figure 22. Typical gas velocity distribution at the inlet to a baghouse.*

## 2.3 PARTICLE SIZE MEASUREMENT TECHNIQUES

### 2.3.1 General Discussion

Any detailed experimental program designed to evaluate fabric filters must include measurements of the particle size distributions at the inlet and outlet. These size distributions can then be used to calculate the baghouse collection efficiency versus particle size, or "fractional efficiency curve".

Although most of the mass emitted from a particular pollution source may consist of large particles, in general, the largest number of particles is in the fine particle range. Thus, high mass collection efficiency does not always imply high number collection efficiency nor does it insure that a particular opacity standard will be met. Fine particles also contribute more to visible light scattering and opacity and present a greater health hazard than do the larger particles.

An ideal particle size measurement device would be located in situ and give a real time readout of particle size distributions and particle number concentration over the size range from 0.01 μm to 10 μm diameter. At the present time, however, particle size distribution measurements are made using several instruments which operate over limited size ranges and do not yield instantaneous data.

Particle sizing methods may involve instruments which are operated in-stack, or out of stack where the samples are taken using probes. For in-stack sampling, the sample aerosol flow rate is usually adjusted to maintain near isokinetic sampling conditions in order to avoid concentration errors which result from under or oversampling large particles (dia. > 3 μm) which have too high an inertia to follow the gas flow streams in the vicinity of the sampling nozzle. Since many particulate sizing devices have size fractionation points that are flow rate dependent, the necessity for isokinetic sampling in the case of large particles can result in undesirable compromises in obtaining data - either in the number of points sampled or in the validity or precision of the data for large particles.

In general, particulate concentrations within a duct or flue are stratified to some degree with strong gradients often found for larger particles and in some cases for small particles. Such concentration gradients, which can be due to inertial effects, gravitational settling, leaking compartments, etc., imply that multipoint (traverse) sampling must be used. Even the careful use of multipoint traverse techniques will not guarantee that representative data are obtained. The location of the sampling points during process changes or variations in baghouse operation can lead to significant scatter in the data.

Choices of particulate measurement devices or methods for individual applications are dependent on the availability of suitable techniques which permit the required temporal and/or spatial resolution or integration. In many instances the properties of the particulate are subject to large changes in not only size distribution and concentration, but also in chemical composition (for example, emissions from the open hearth steel making process). Different methods or sampling devices are generally required to obtain data for long term process averages as opposed to the isolation of certain portions of the process in order to determine the cause of a particular type of emission.

Interferences exist which can affect most sampling methods. Two commonly occurring problems are the condensation of vapor phase components from the gas stream and reactions of gas, liquid, or solid phase materials with various portions of the sampling systems. An example of the latter is the formation of sulfates in appreciable (several milligram) quantities on several of the commonly used glass fiber filter media by reactions involving $SO_x$ and trace constituents of the filter media. Sulfuric acid condensation in cascade impactors and in the probes used for extractive sampling is an example of the former.

If extractive sampling is used and the sample is conveyed through lengthy probes and transport lines, as is the case with several particulate sizing methods, special attention must be given toward recognition, minimization, and compensation for losses by various mechanisms in the transport lines. The degree of such losses can be quite large for certain particle sizes.

In this section, individual particulate sampling systems and procedures are discussed. These are categorized according to the physical mechanism that is used to obtain the data: inertial, optical, electrical, or diffusional.

### 2.3.2 Inertial Particle Sizing Devices

Two devices which fall into the inertial sizing category are impactors and cyclones. In both of these devices, the aerosol stream is constrained to follow a path of such curvature that the particles tend to move radially outward toward a collection surface because of their inertia. Subsequent analysis of the particle size distribution may be made by gravimetric means, quantitative chemical analysis, or microscopic inspection.

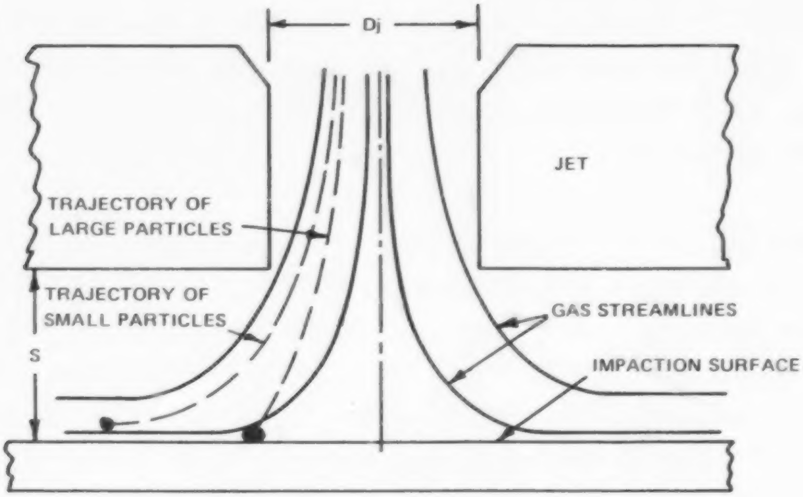
Particle size distribution measurements related to fabric filter evaluations have largely been made using cascade impactors, which are effective in the size range from 0.3 to 20 μm diameter; although, in some cases, hybrid cyclone-impactor units, or cyclones have also been used. The particle size distributions are normally calculated from experimental data by relating the mass collected on various stages to the theoretical or calibrated size cutpoints associated with those stage geometries.

Cascade impactors -- Because of its compact arrangement and mechanical simplicity, the cascade impactor has gained wide acceptance as a practical means of making particle size measurements in flue gases. In most cases, the impactors can be inserted directly into the duct or flue, eliminating many condensation and sample loss problems which occur when probes are used for extractive sampling.

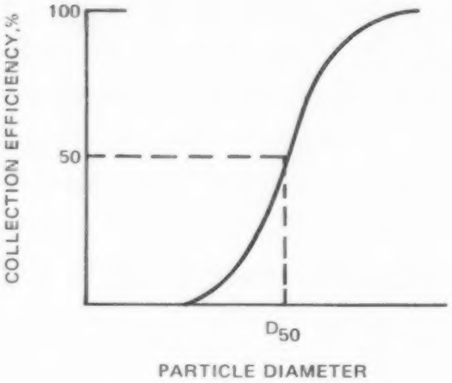
Figure 24 is a schematic which illustrates the principle of particle collection which is common to all cascade impactors. The sample aerosol is constrained to pass through a slit or circular hole to form a jet which is directed toward an impaction surface. Particles which have lower momentum will follow the air stream to lower stages where the jet velocities are progressively higher. For each stage there is a characteristic particle size which theoretically has a 50% probability of striking the collection surface. This particle size, or $D_{50}$, is called the effective cut size for that stage. The number of holes or jets on any one stage ranges from one to several hundred depending on the desired jet velocity and total volumetric flow rate. The number of jet stages in an impactor ranges from one to about twenty for various impactor geometries reported in the literature. Most commercially available impactors have between five and ten stages.

Parameters which determine the collection efficiency for a particular geometry are the gas viscosity, the particle density, the jet diameter or width, the jet-to-plate spacing, and the velocity of the air jet.

Most modern impactor designs are based on the semi-empirical theory of Ranz and Wong (1952). More comprehensive theories have been developed by Davies and Aylward (1951) and by Marple (1970). In practice, deviations from ideal behavior in actual impactors dictate that they be calibrated experimentally.


Dj
JET
TRAJECTORY OF
LARGE PARTICLES
TRAJECTORY OF
SMALL PARTICLES
S
GAS STREAMLINES
IMPACTION SURFACE

**A. TYPICAL IMPACTOR JET AND COLLECTION PLATE**



**B. GENERALIZED STAGE COLLECTION EFFICIENCY CURVE**

3630-059

*Figure 23. Operation principle and typical performance for a cascade impactor.*

A large number of experimental studies have been published on cascade impactor design and performance in the laboratory environment. Most of these have been reviewed in the dissertations of Marple (1970) and Rao (1975). Recently, Cushing et al (1976) have presented calibration data on several commercially available cascade impactors. Figures 25, 26, 27, 28, and 29 show schematics of the commercial impactor designs which are commonly used in source testing. Table III gives a listing of the manufacturers, and some operational information for stack sampling. The details of cascade impactor applications are discussed in Appendix C.

It is usually impractical to use the same impactor at the inlet and outlet of a fabric filter when making fractional efficiency measurements because of the large difference in particulate loading. For example, if a sampling time of thirty minutes is adequate at the inlet, for the same impactor operating conditions and the same amount of sample collected, approximately 3000 minutes sampling time would be required at the outlet (a collection efficiency of 99% is assumed). Although impactor flow rates can be varied, they cannot be adjusted enough to compensate for this difference in particulate loading without creating other problems. Extremely high sampling rates result in particle bounce and in scouring of impacted particles from the lower stages of the impactor where the jet velocities become excessively high. Short sampling times may result in atypical samples being obtained as a result of momentary fluctuations in the particle concentration or size distribution within the duct. Normally, a low flow rate impactor is used at the inlet and a high flow rate impactor at the outlet. The impactors are then operated at their respective optimum flow rates, and the sampling times are dictated by the time required to collect weighable samples on each stage without overloading any single stage.

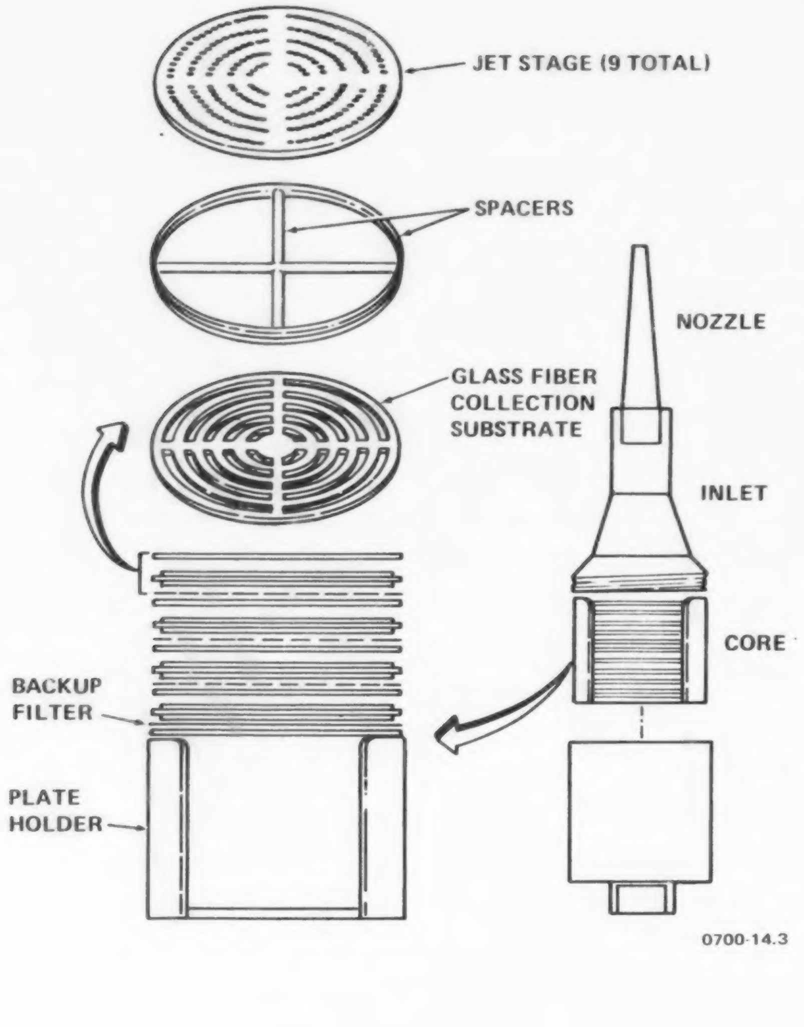

JET STAGE (9 TOTAL)
SPACERS
NOZZLE
GLASS FIBER COLLECTION SUBSTRATE
INLET
CORE
BACKUP FILTER
PLATE HOLDER
0700-14.3

*Figure 24. Andersen Mark III Stack Sampler.*

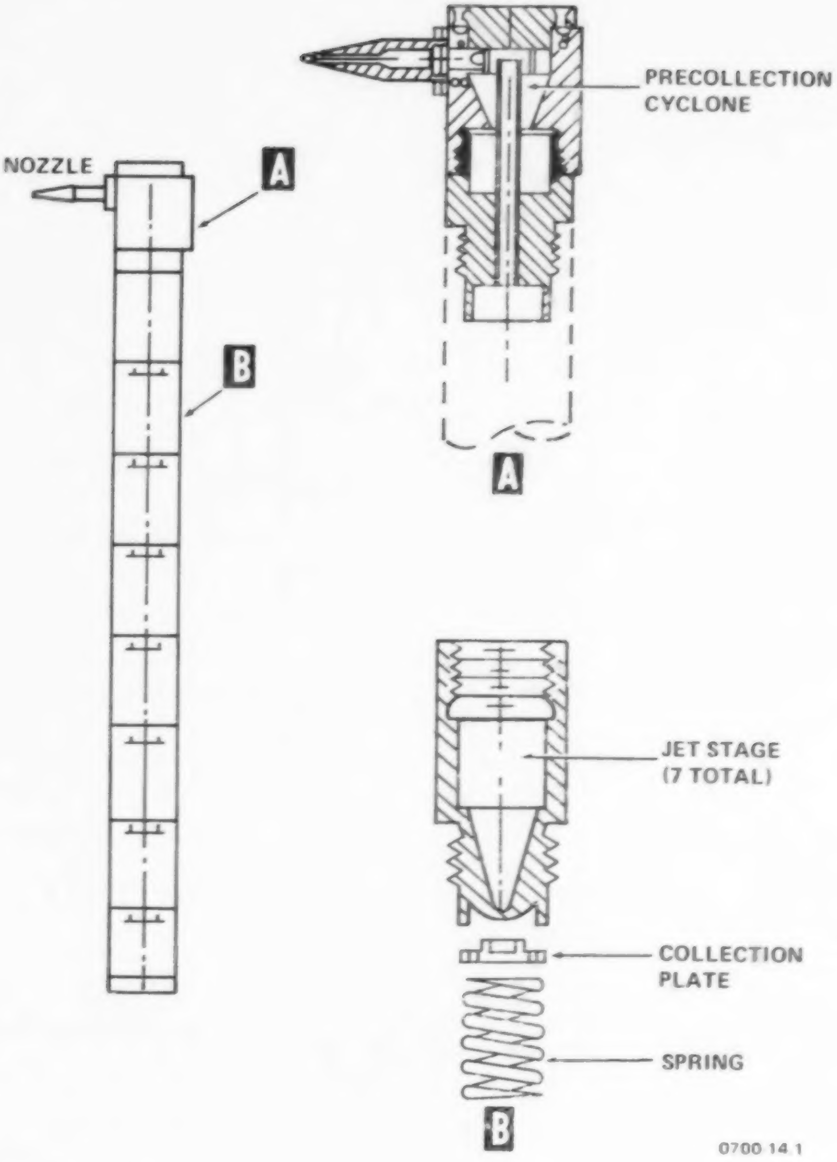

NOZZLE
A
B
PRECOLLECTION CYCLONE
A
JET STAGE (7 TOTAL)
COLLECTION PLATE
SPRING
B
0700 14.1

*Figure 25. Modified Brink Model BMS-11 Cascade Impactor.*

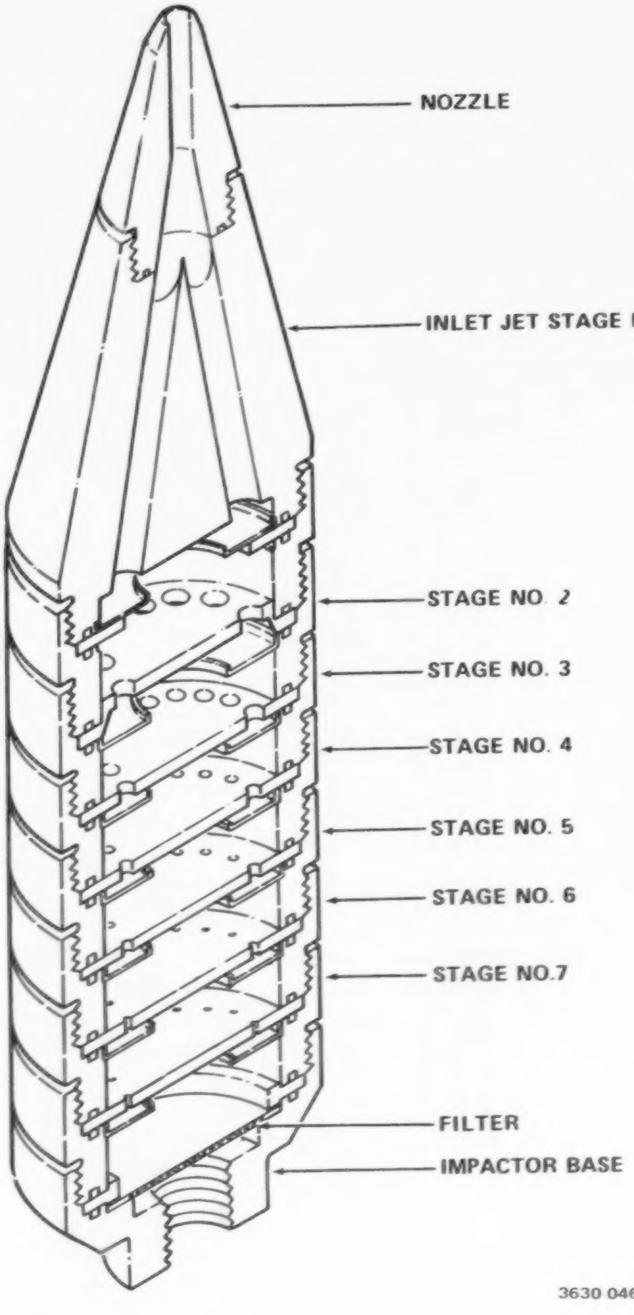

NOZZLE
INLET JET STAGE NO 1
STAGE NO. 2
STAGE NO. 3
STAGE NO. 4
STAGE NO. 5
STAGE NO. 6
STAGE NO.7
FILTER
IMPACTOR BASE
3630 046

*Figure 26. MRI Model 1502 Inertial Cascade Impactor.*

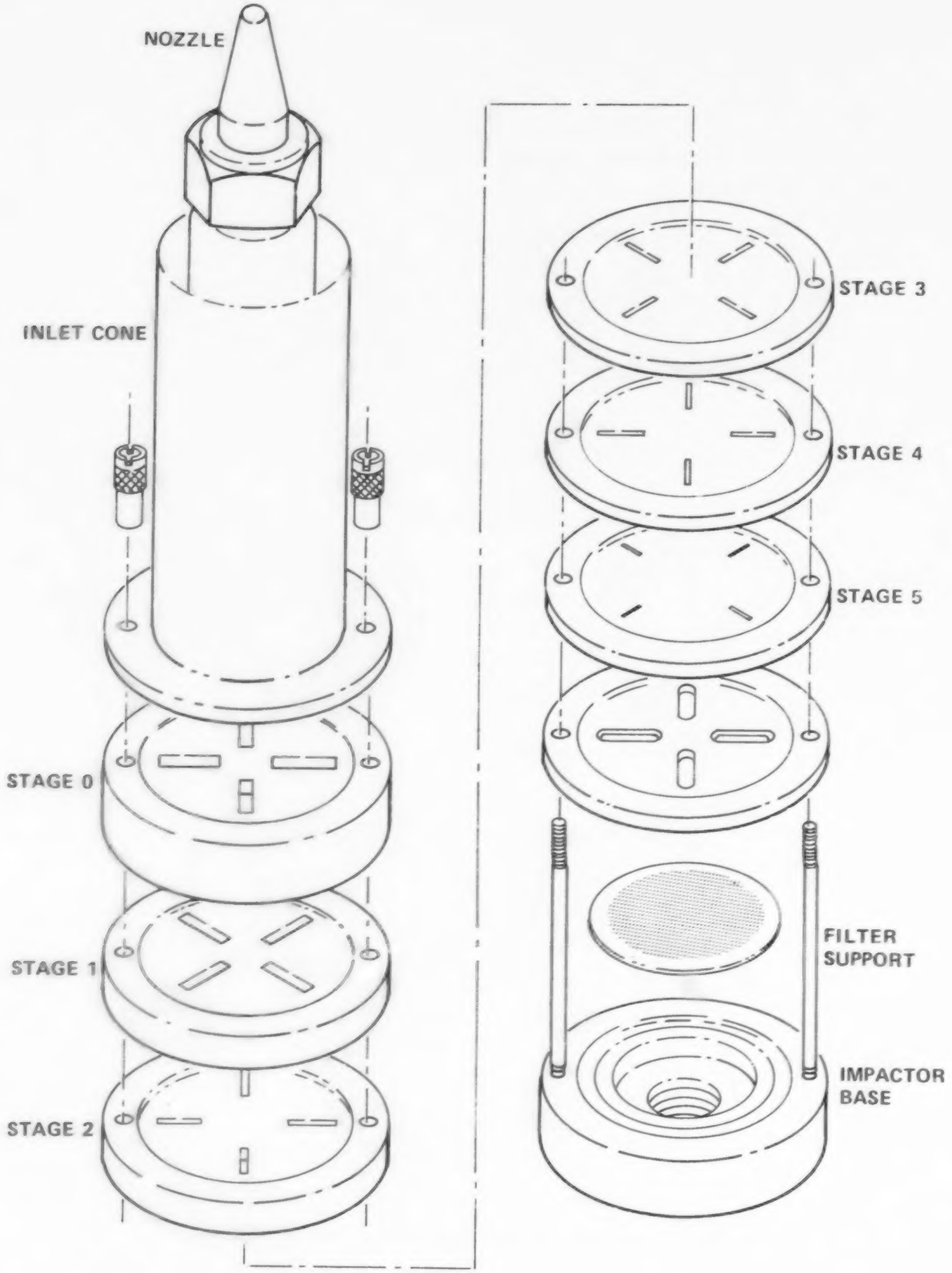

NOZZLE
INLET CONE
STAGE 0
STAGE 1
STAGE 2
STAGE 3
STAGE 4
STAGE 5
FILTER SUPPORT
IMPACTOR BASE
3630-053

*Figure 27. Sierra Model 226 Cascade Impactor.*

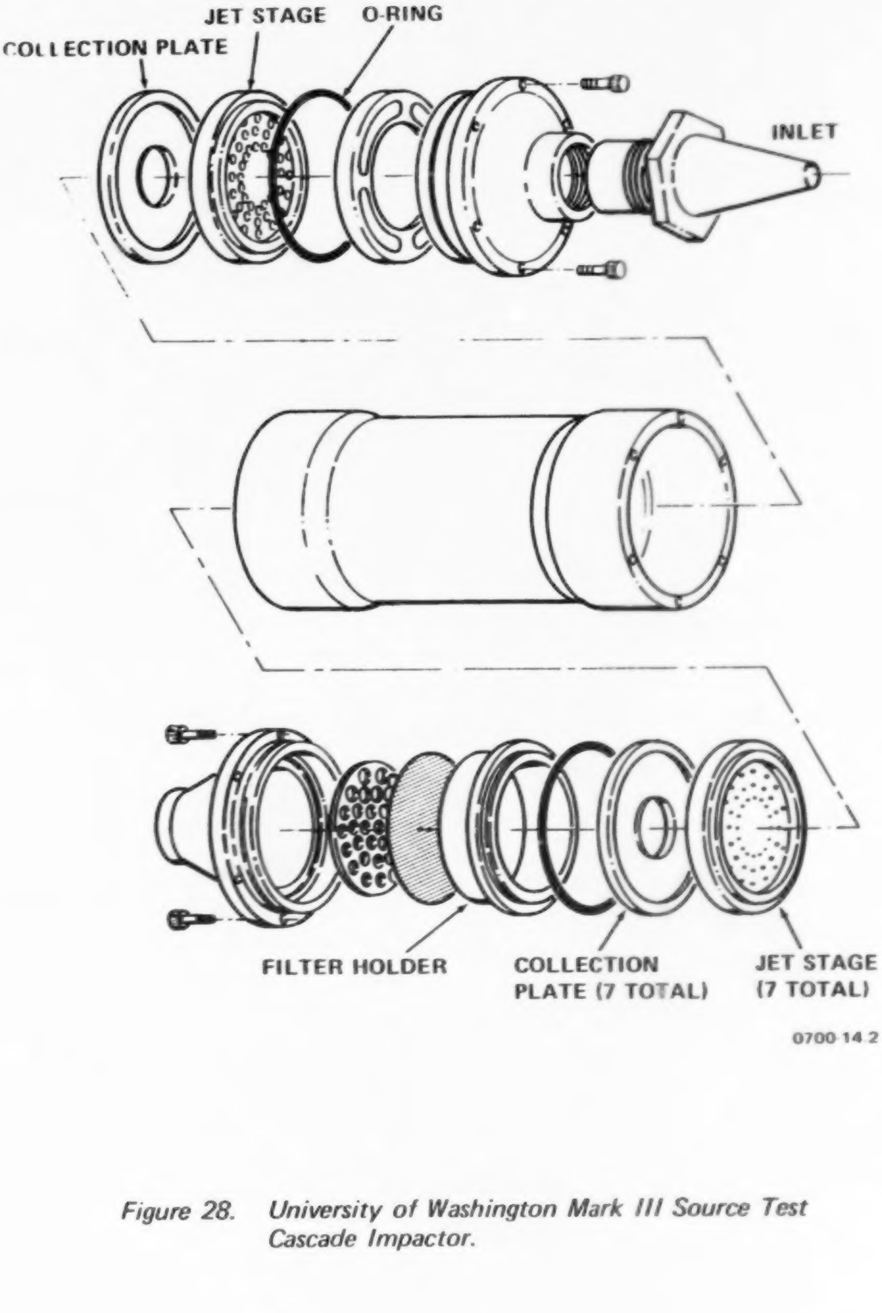

JET STAGE
O-RING
COLLECTION PLATE
INLET
FILTER HOLDER
COLLECTION PLATE (7 TOTAL)
JET STAGE (7 TOTAL)
0700-14.2

*Figure 28. University of Washington Mark III Source Test Cascade Impactor.*

## TABLE III

## COMMERCIAL CASCADE IMPACTOR SAMPLING SYSTEMS

| Name | Nominal Flow rate (cm$^3$/sec) | Substrates | Manufacturer |
|---|---|---|---|
| Andersen Stack Sampler (Precollection Cyclone Avail.) | 236 | Glass Fiber (Available from manufacturer) | Andersen 2000, Inc.<br>P.O. Box 20769<br>Atlanta, GA 30320 |
| Univ. of Washington Mark III Source Test Cascade Impactor (Precollection Cyclone Avail.) | 236 | Stainless Steel Inserts, Glass Fiber, Grease | Pollution Control System Corp.<br>321 Evergreen Bldg.<br>Renton, WA 98055 |
| Brink Cascade Impactor (Precollection Cyclone Avail.) | 14.2 | Glass Fiber, Aluminum, Grease | Monsanto EnviroChem Systems, Inc.<br>St. Louis, MO 63166 |
| Sierra Source Cascade Impactor - Model 226 (Precollection Cyclone Avail.) | 118 | Glass Fiber (Available from manufacturer) | Sierra Instruments, Inc.<br>P.O. Box 909<br>Village Square<br>Carmel Valley, CA 93924 |
| MRI Inertial Cascade Impactor | 236 | Stainless Steel, Aluminum, Mylar, Teflon. Optional: Gold, Silver, Nickel | Meteorology Research, Inc.<br>Box 637<br>Altadena, CA 91001 |

Series cyclone particulate sampling techniques -- Prototype series cyclone sampling systems have been developed for industrial source sampling (Chang, 1974). In general, series cyclones are easy to use, trouble-free, and efficient collectors of large quantities of size segregated particulate. Their main drawbacks are that their size limits the number of size segregated samples during each test that can be obtained as compared to most commercial impactors and an accurate theory of operation has not been developed. However, the ability to collect large quantities of sized material for analysis makes these devices irreplaceable for some applications.

The Source Assessment Sampling System (SASS) incorporates three cyclones and a back up filter (Blake, 1978). A schematic of this system is illustrated in Figure 30. It is operated at a flow rate of 3065 $cm^3$/sec (6.5 $ft^3$/min) with approximate cyclone cut points of 10, 3, and 1 micrometer aerodynamic diameter and a gas temperature of 205°C. Besides obtaining information on the particle size distribution, this system collects gram quantities of particles for later chemical or biological analyses. The SASS train is large and requires extractive sampling through a heated probe. The cyclones are mounted in an oven to keep the air stream at stack temperature or above the dew point until the particles are collected. This system is supplied with nozzles, a probe-pitot-tube-thermocouple assembly, cyclones, back up filter, oven, a gas conditioning chamber, and a flow metering device and pump adapted from the Aerotherm High Volume Sampling System. Cyclone calibration details are furnished along with equations for calculating approximate cyclone cutpoints for operating conditions other than those measured during the calibration procedure.

It is mandatory that the gas velocity and temperature through the cyclones be maintained at a constant setting while sampling, because the cyclone cut points are dependent upon the gas flow rate and temperature. This usually means that periods of non-

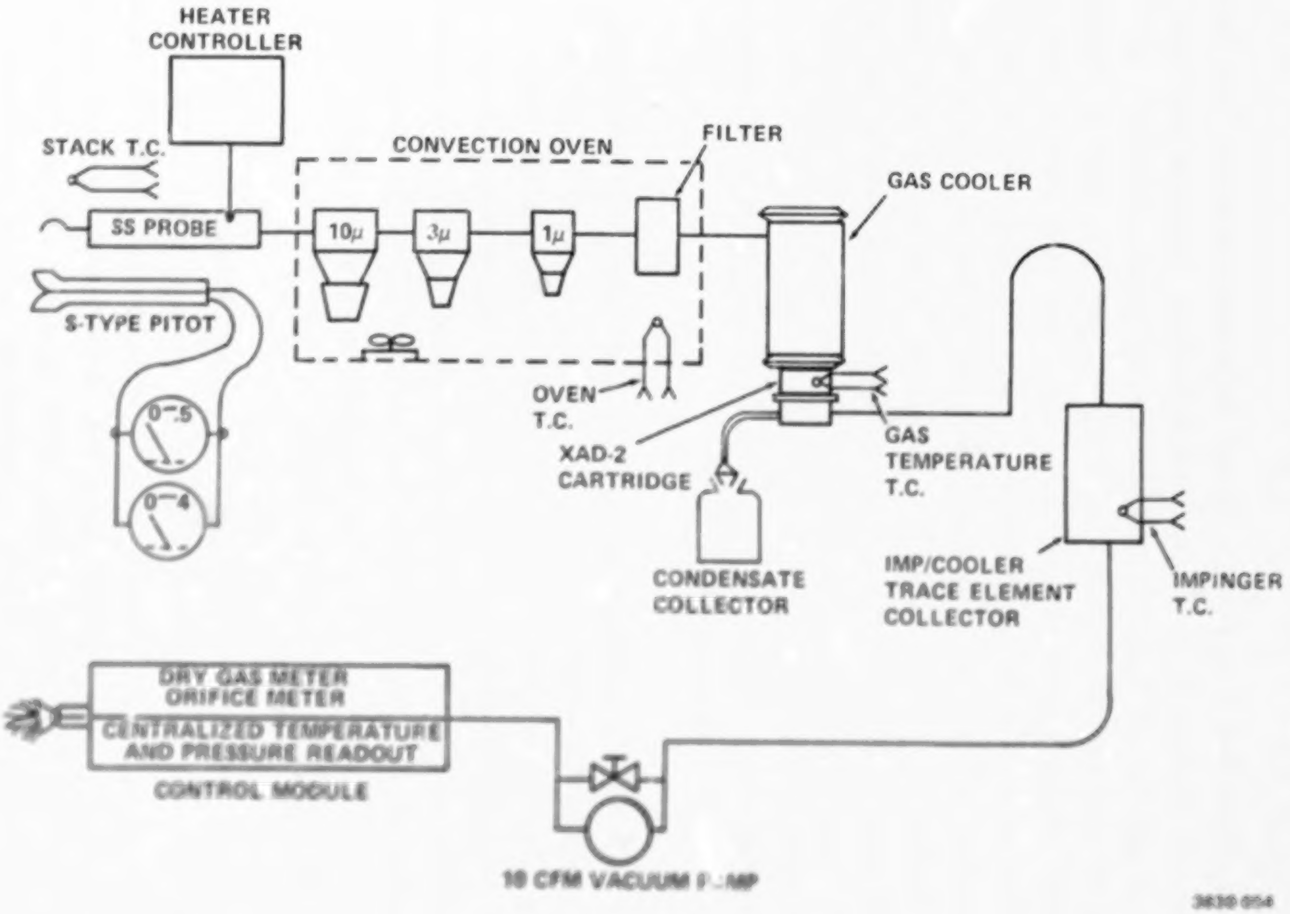

HEATER CONTROLLER
STACK T.C.
SS PROBE
S-TYPE PITOT
0–.5
0–4
CONVECTION OVEN
10μ
3μ
1μ
FILTER
GAS COOLER
OVEN T.C.
XAD-2 CARTRIDGE
CONDENSATE COLLECTOR
GAS TEMPERATURE T.C.
IMP/COOLER TRACE ELEMENT COLLECTOR
IMPINGER T.C.
DRY GAS METER
ORIFICE METER
CENTRALIZED TEMPERATURE AND PRESSURE READOUT
CONTROL MODULE
10 CFM VACUUM PUMP
3830 054

Figure 29. Schematic of the Source Assessment Sampling System.

isokinetic sampling may occur. Depending on magnitude of fluctuations in the velocity of the sampled stream, this may or may not introduce significant errors in the sizing process.

Southern Research Institute, under EPA sponsorship, has designed and built a prototype three-stage series cyclone for in-stack use. A sketch of this system is shown in Figure 31. It is designed to operate at 472 $cm^3$/sec (1 $ft^3$/min). The calibrated cut points for these cyclones are 3.0, 1.6, and 0.6 micrometer aerodynamic at 21°C. A 47 mm Gelman filter holder is used as a back up filter after the last cyclone. This series cyclone system was designed for in-stack use and requires a six inch sampling port. A sampling system similar to that for a high flow rate impactor is usually adequate, although a more powerful pump may be required under some sampling situations. As with the SASS train, a constant flow rate through the cyclone system is required to maintain stable cyclone cut points.

Figure 32 shows a second generation EPA/Southern Research series cyclone system under development which contains five cyclones and a back up filter. It is a compact system and will fit through 4 inch diameter ports. The initial prototype was made of anodized aluminum with stainless steel connecting hardware. A second prototype, for in-stack evaluation, is made of titanium. The development of this cyclone system is summarized in a report by Smith and Wilson (1978).

Figure 33 shows laboratory calibrations of the five cyclones in the prototype system. The cut points, at the test conditions are 0.32, 0.6, 1.3, 2.6, and 7.5 μm. A continuing research program includes studies to investigate the dependence of the cyclone cut points upon the sample flow rate and temperature so that the behavior of the cyclones at stack conditions can be predicted more accurately.

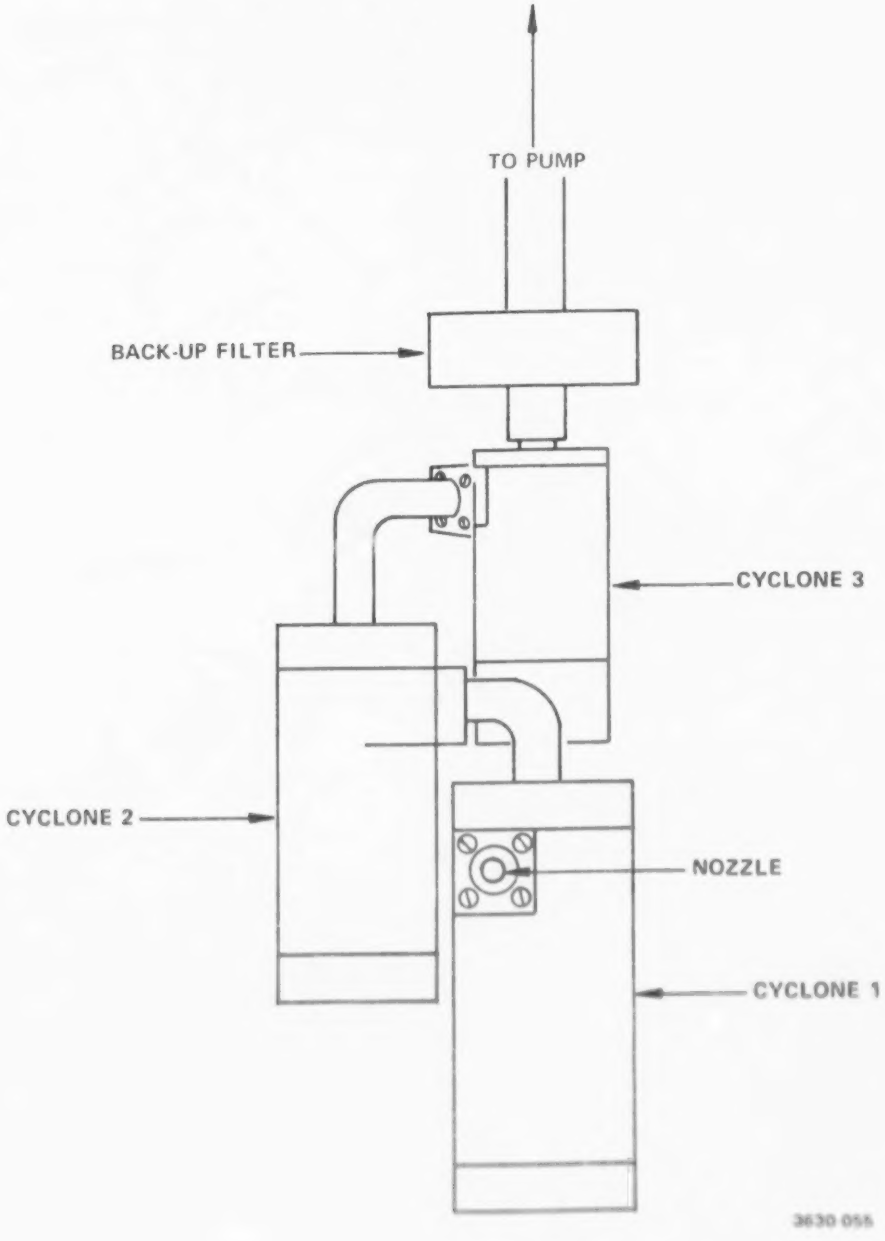

TO PUMP
BACK-UP FILTER
CYCLONE 3
CYCLONE 2
NOZZLE
CYCLONE 1
3630 055

*Figure 30. Three Stage Series Cyclone System.*

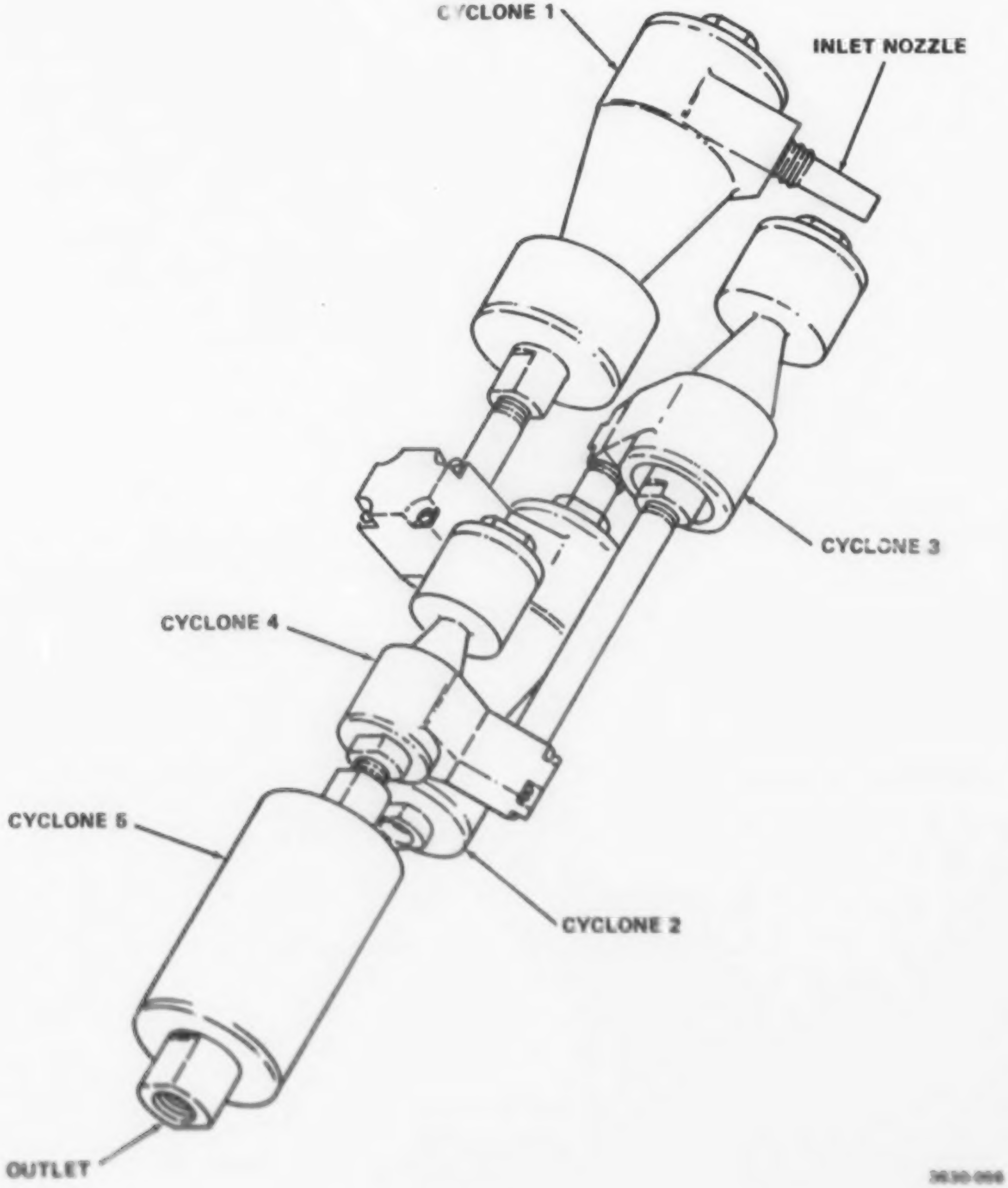

CYCLONE 1
INLET NOZZLE
CYCLONE 3
CYCLONE 4
CYCLONE 5
CYCLONE 2
OUTLET
3630-066

*Figure 31. Five Stage Series Cyclone System.*

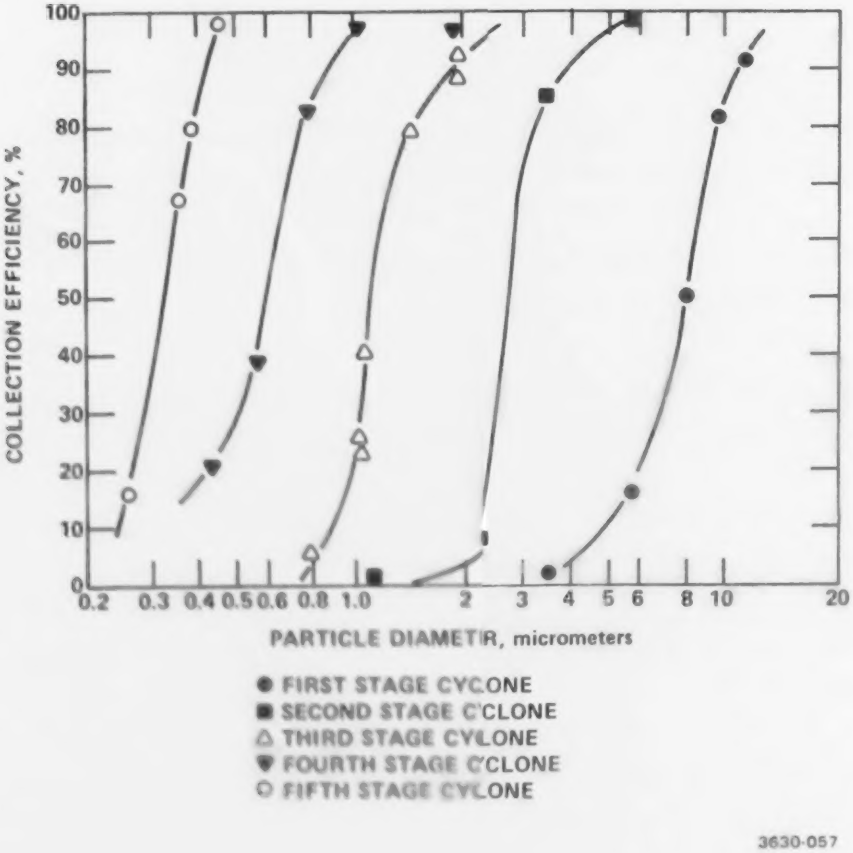


*Figure 32. Laboratory Calibration for the Five Stage Series Cyclone System. (472 cm/sec, particle density—1.0 gm/cm3)*

Small cyclone systems appear to be practical alternatives to cascade impactors as instruments for measuring particle size distributions in process streams. Cyclones offer several advantages:

Large, size segregated samples are obtained.

There are no substrates to interfere with analyses.

They are convenient and reliable to operate.

They allow long sampling times under high mass loading conditions for a better process emission average.

They may be operated at a wide range of flow rates without particle bounce or reentrainment.

On the other hand, there are some negative aspects of cyclone systems which require further investigation:

Unduly long sampling times may be required to obtain large samples at relatively clean sources.

The existing theories do not accurately predict cyclone performance.

Cyclone systems are bulkier than impactors and may require larger ports for in-stack use.

As discussed above, cyclones are now used on an experimental basis by the EPA and EPA contractors. If current research programs are successful in developing a better understanding of cyclone behavior, they may play a very important role in control device evaluation.

### 2.3.3. Optical Measurement Techniques

The basic operating principle for one type of optical particle counter is illustrated in Figure 34. Light is scattered by individual particles as they pass through a small viewing volume, the intensity of the scattered light being measured by a photodetector. The sizes of the particles determine the amplitude of the scattered light pulses, and the rate at which the pulses occur is related to the particle concentration. Thus, a counter of this type gives both size and concentration information. The simultaneous presence of more than one particle in the viewing volume is interpreted by the counter as a larger single particle. To avoid errors arising from this effect, dilution to about 300 particles/cm$^3$ is generally necessary. Errors in counting rate also occur as a result of electronics deadtime and from statistical effects resulting from the presence of high concentrations of sub-countable ($D < 0.3$ μm) particles in the sample gas stream (Whitby and Liu, 1967). The intensity of the scattered light depends upon the viewing angle, particle index of refraction, particle optical absorptivity, and shape, in addition to the particle size. The schematic in Figure 34 shows a system which utilizes "integrated near forward" scattering. Different viewing angles might be chosen to optimize some aspect of the counter performance. For example, near forward scattering minimizes the affect of variations in the indicated particle size with index of refraction, but for this geometry, there is a severe loss of resolution for particle diameters near 1 μm. Right angle, or 90° scattering smooths out the response curve, but the intensity is more dependent on the particle index of refraction.

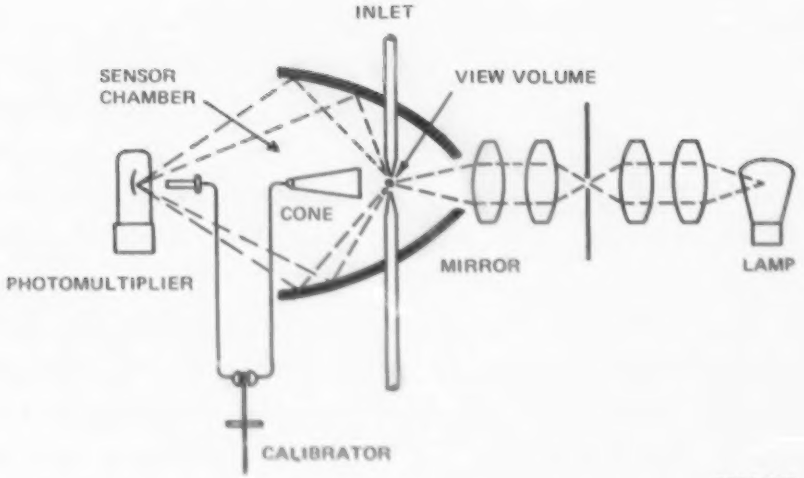

INLET
SENSOR CHAMBER
VIEW VOLUME
CONE
PHOTOMULTIPLIER
MIRROR
LAMP
CALIBRATOR
3630-060

*Figure 33. Operating principle for an optical particle counter. Courtesy of Climet Instruments Company.*

Available geometries are:

| | |
|---|---|
| Bausch & Lomb 40-1 | Near Forward Scattering |
| Royco 220 | Right Angle |
| Royco 245, 225 | Near Forward |
| Climet CI-201, 208 | Integrated Near Forward |

Optical particle counters have not been used extensively in stack sampling because they cannot be applied directly to the effluent gas stream. The sample must be extracted, cooled, and diluted; a procedure which requires great care to avoid introducing serious errors into calculations of the particle size distribution. The main advantage of optical counters is the capability of observing emission fluctuations in real time. After extraction, the useful particle size range is approximately 0.3 to 1.5 μm.

### 2.3.4 Ultrafine Particle Sizing Techniques

There are two physical properties of ultrafine particles (diameter < 0.5 μm) which are size dependent and which can be predicted with sufficient accuracy under controlled conditions to be used to measure particle size. These are the particle diffusivity and electrical mobility. Although ultrafine particle size distribution measurements are still in a developmental stage, instruments are available which can be used for this purpose, and some field measurements have been made. A practical limitation on the lower size limit for this type of measurement is the loss of particles by diffusion in the sampling lines and instrumentation. These losses are excessive for particle sizes below about 0.01 μm where the samples are extracted from a duct and diluted to concentrations within the capability of the sensing devices.

Diffusional sizing -- Diffusion batteries may consist of a number of long, narrow, parallel channels, a cluster of small bore tubes, or a series of screens. Figure 35a shows a typical parallel channel diffusion battery, and Figure 35b shows the aerosol penetration characteristics of this geometry at two flow rates. The parallel plate geometry is convenient because of ease of fabrication and the availability of suitable materials, and also because sedimentation can be ignored if the slots are vertical, while additional information can be gained through settling, if the slots are horizontal.

Breslin et al (1971) and Sinclair (1972) report success with more compact, tube-type and screen-type arrangements in laboratory studies and a commercial version of Sinclair's geometry is available.* Although the screen-type diffusion battery must be calibrated empirically, it offers convenience in cleaning and operation, and compact size. Figure 36 shows Sinclair's geometry. This battery is 21 cm long, approximately 4 cm in diameter, and weighs 0.9 kg.

Variations in the length and number of channels (tubes, or screens) and in the aerosol flow rate are used as means of measuring the number of particles in a selected size range. As the aerosol moves in streamline flow through the channels, the particles diffuse to the walls at a predictable rate, depending on the particle size and the diffusion battery geometry. It is assumed that every particle which reaches the battery wall will adhere, therefore, only a fraction of the influent particles will appear at the effluent of a battery. It is only necessary to measure the total number concentration of particles with a condensation nuclei counter at the inlet and outlet to the diffusion battery under a number of conditions in order to calculate the particle size distribution.

---

*TSI Incorporated, 500 Cardigan Road, St. Paul, MN 55165.

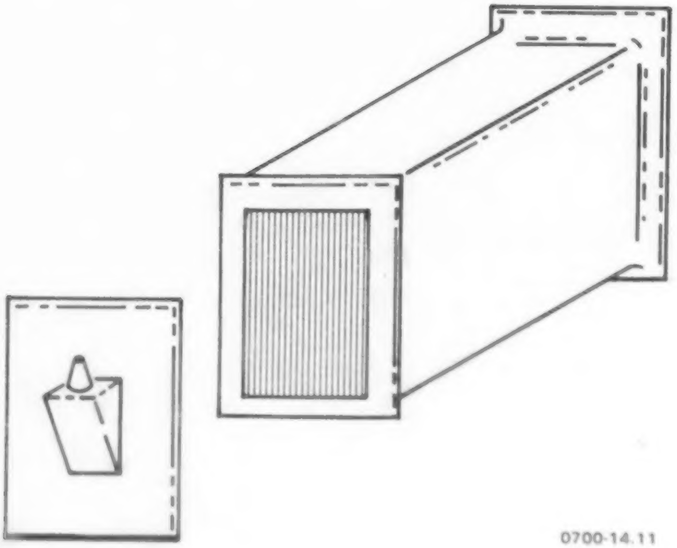


*Figure 34a. Parallel plate diffusion battery.*

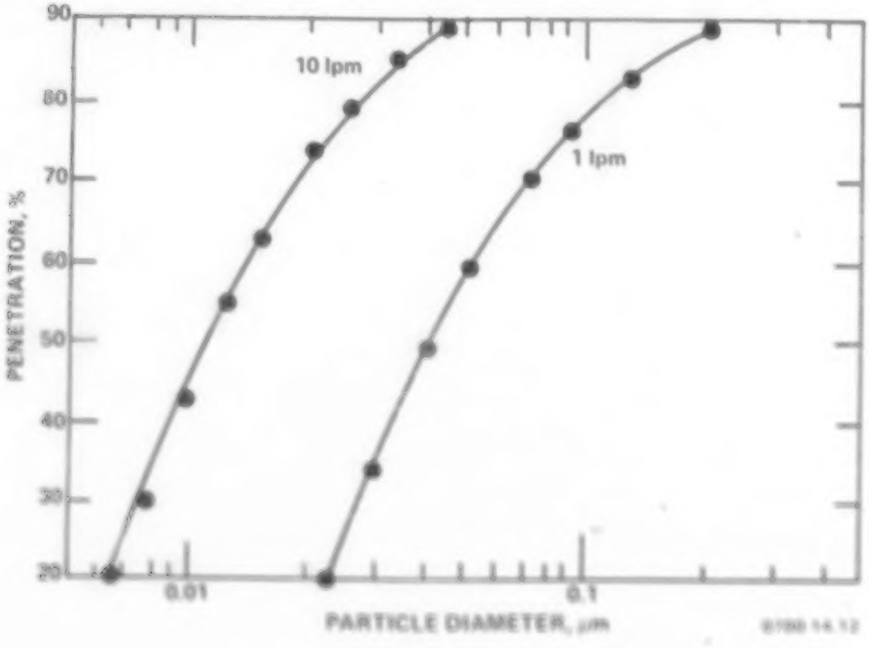


*Figure 34b. Parallel plate diffusion battery penetration curves for monodisperse aerosols (12 channels, 0.1 × 10 × 48 cm).*

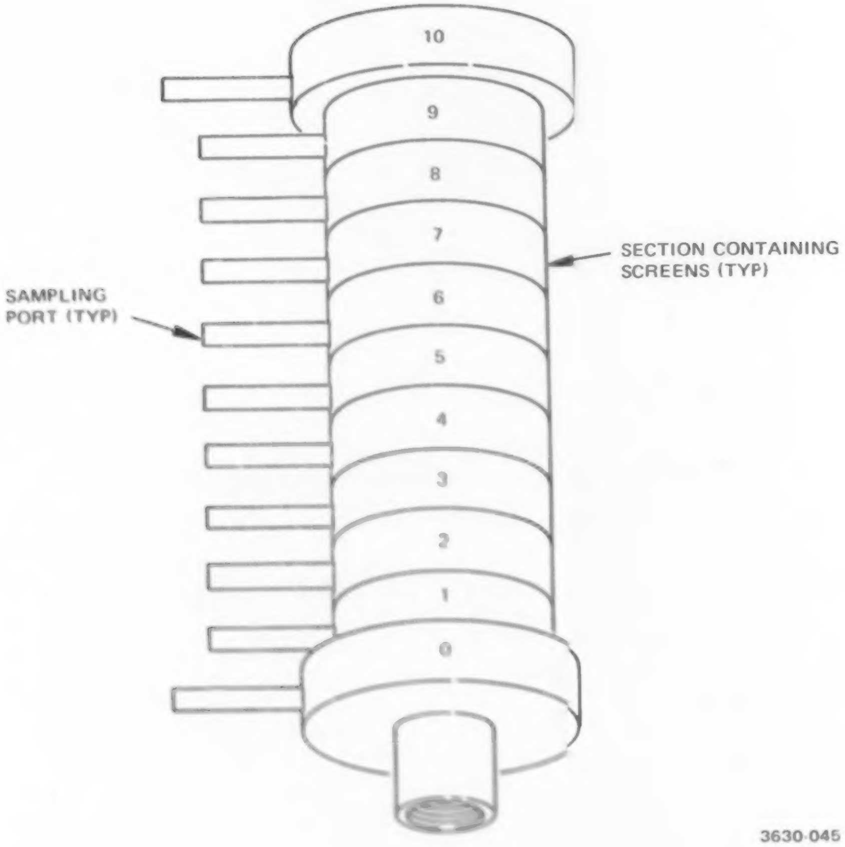

10
9
8
7
6
5
4
3
2
1
0
SECTION CONTAINING SCREENS (TYP)
SAMPLING PORT (TYP)
3630-045

*Figure 35. Screen type diffusion battery. The battery is 21 cm long, 4 cm in diameter, and contains 55, 635 mesh stainless steel screens.*

Diffusional measurements are less dependent upon the aerosol parameters than the other techniques discussed and perhaps are on a more firm basis from a theoretical standpoint.

Disadvantages of the diffusional technique are the bulk of the parallel plate diffusional batteries, although advanced technology may alleviate this problem; the long time required to measure a size distribution; and problems with sample conditioning when condensible vapors are present.

Electrical particle counters -- Several aerosol spectrometers, or mobility analyzers, have been demonstrated that employ the diameter-mobility relationship and to classify particles according to their size, and Figure 36 illustrates the principle on which these devices operate. Particles are charged under conditions of homogeneous electric field and ion concentration, and then passed into the spectrometer. Clean air flows down the length of the device and a transverse electric field is applied. From a knowledge of the system geometry and operating conditions, the mobility is derived for any position of deposition on the grounded electrode. The particle diameter is then readily calculated from a knowledge of the electric charge and mobility.

Difficulties with mobility analyzers are associated primarily with charging the particles to a known value with a minimum of loss by precipitation and obtaining accurate analyses of the quantity of particles in each size range. The latter may be done gravimetrically, optically, or electrically.

The concept described above has been used by Liu, Whitby, and Pui (1974) at the University of Minnesota, to develop a series of Electrical Aerosol Analyzers (EAA). A commercial version of the U. of Minnesota devices is now marketed by Thermosystems, Inc. as the Model 3030 (Figure 37).* The EAA is designed to measure the

---

*TSI Incorporated, 500 Cardigan Road, St. Paul, MN 55165.

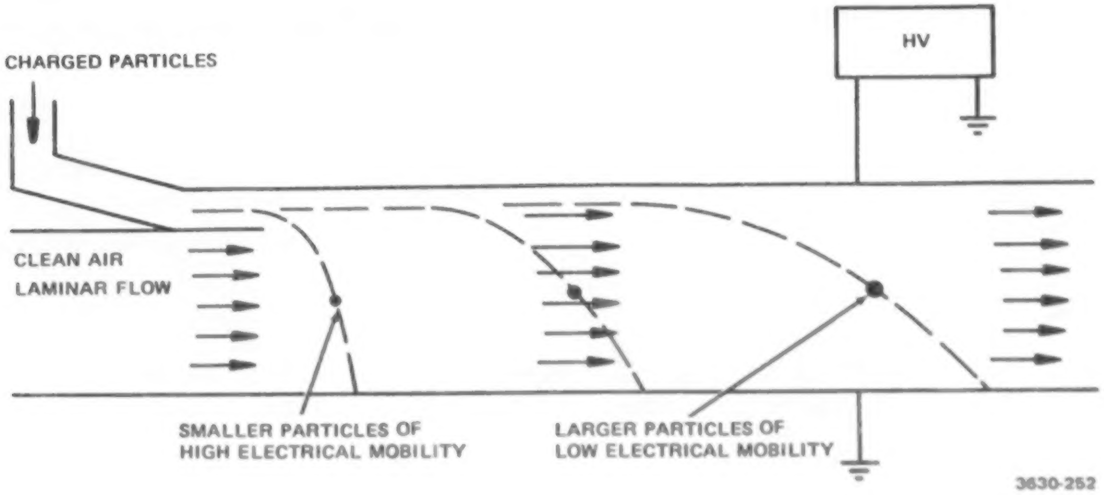

CHARGED PARTICLES
HV
CLEAN AIR
LAMINAR FLOW
SMALLER PARTICLES OF
HIGH ELECTRICAL MOBILITY
LARGER PARTICLES OF
LOW ELECTRICAL MOBILITY
3630-252

*Figure 36. The electric mobility analyzer principle.*

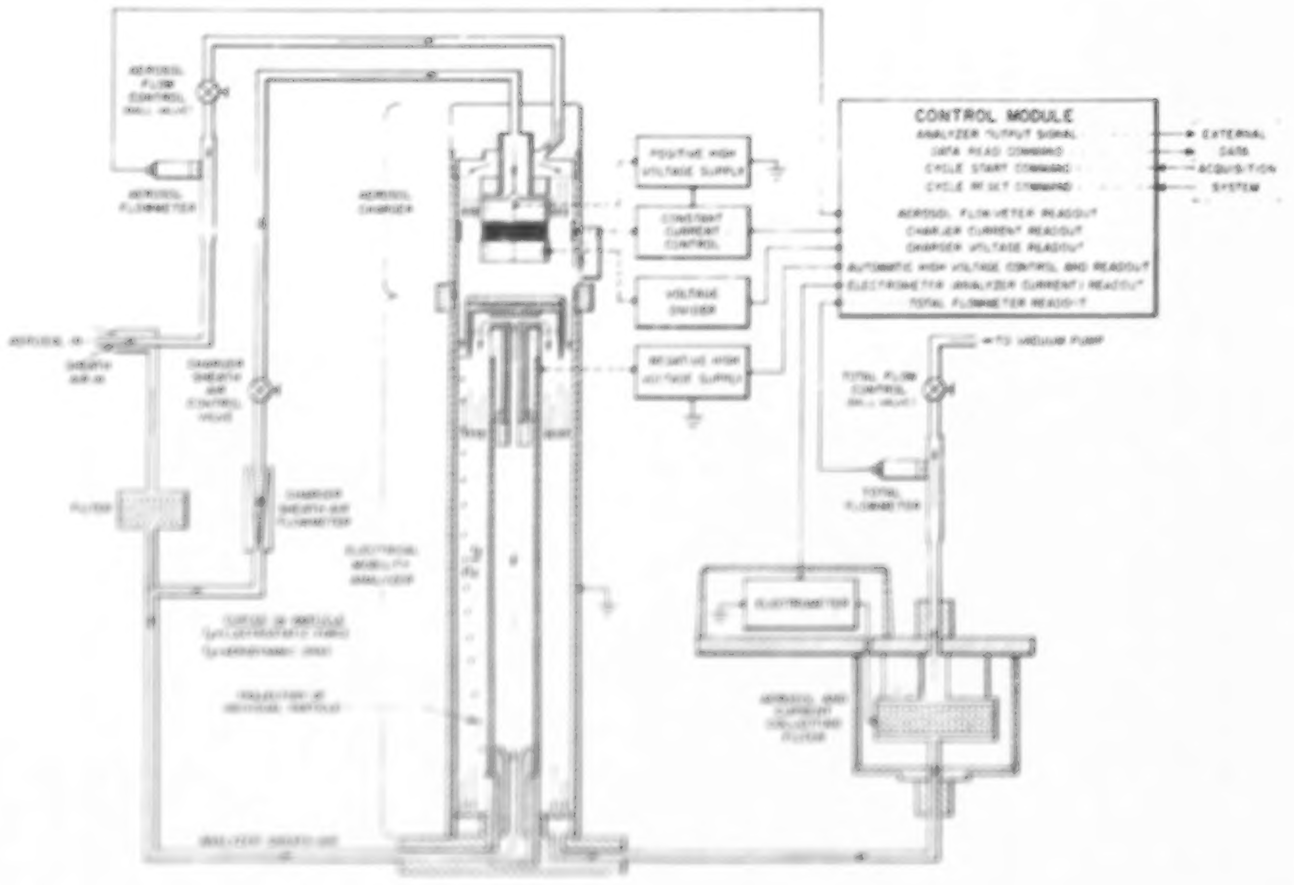

AEROSOL FLOW CONTROL BALL VALVE
AEROSOL FLOWMETER
AEROSOL IN
SHEATH AIR IN
CHARGER SHEATH AIR CONTROL VALVE
FILTER
CHARGER SHEATH AIR FLOWMETER
AEROSOL CHARGER
ELECTRICAL MOBILITY ANALYZER
ANALYZER SHEATH AIR
POSITIVE HIGH VOLTAGE SUPPLY
CONSTANT CURRENT CONTROL
VOLTAGE DIVIDER
NEGATIVE HIGH VOLTAGE SUPPLY
CONTROL MODULE
ANALYZER OUTPUT SIGNAL
DATA READ COMMAND
CYCLE START COMMAND
CYCLE RESET COMMAND
AEROSOL FLOWMETER READOUT
CHARGER CURRENT READOUT
CHARGER VOLTAGE READOUT
AUTOMATIC HIGH VOLTAGE CONTROL AND READOUT
ELECTROMETER (ANALYZER CURRENT) READOUT
TOTAL FLOWMETER READOUT
EXTERNAL DATA ACQUISITION SYSTEM
TO VACUUM PUMP
TOTAL FLOW CONTROL BALL VALVE
TOTAL FLOWMETER
ELECTROMETER
AEROSOL AND CURRENT COLLECTING FILTER

Figure 32. Flow schematic and electronic block diagram of the Electrical Aerosol Analyzer. After Liu, Whitby, and Pui(1974).

size distribution of particles in the range from 0.0032 to 1.0 μm diameter. The concentration range for best operation is 1 to 1000 μg/m$^3$, thus dilution is required for most industrial gas aerosols.

The EAA has the distinct advantage of very rapid data acquisition compared to diffusion batteries and condensation nuclei counters (two minutes as opposed to two hours for a single size distribution analysis.)

Disadvantages of this type of measurement system are difficulties in predicting the particle charge, and the fraction of the particles bearing a charge, with sufficient accuracy, and the requirement for sample dilution when making particle size distribution measurements in flue gases.

The details of sampling with optical particle counters, diffusion batteries with condensation nuclei counters, and EAA using extractive sampling and dilution techniques are presented in Appendix D.

## 2.4 PROCESS EFFLUENT GAS ANALYSIS

### 2.4.1 General Discussion

In evaluating the performance of a fabric filter it is advantageous to know something of the constituents of the effluent gas stream, excluding the particulate matter. Information on gas composition can indicate the potential for future problems with the life of the fabric filter, the integrity of the baghouse structure, or changes in the industrial process. Existence of humidity and temperature near dew points can lead to blinding, caking, and cracking of filter bags, if temperature excusions are likely for the particular process. High $SO_x$ content can cause potential formation of $H_2SO_4$ mists which can ruin bags and baghouses, as well as mask ultrafine particle measurements data. $SO_2$ can also react with certain glass filter substrate media in sampling trains which cause weight changes that mask actual mass collection gains. Also, if chemical agents are being injected to modify the industrial process, or if removal of polluting gases (such as $SO_2$ by Nahcolite injection) is occuring, then measurements of gas composition are necessary.

A gas analysis generally concerns the amount of $N_2$, $O_2$, CO, $CO_2$, and $H_2O$ in the effluent. These can usually be determined using an ORSAT apparatus or one of the EPA Stationary Source Test Methods developed for gas analysis. Studying effluent modification by injection of chemical agents has also resulted in the need for measuring the amounts of other gases such as $NH_3$, $SO_2$, $SO_3$, $NO_x$, etc. These measurement methods are briefly discussed in the following sections.

### 2.4.2 Qualitative Gas Analysis

Interviewing key plant personnel or obtaining the proper records should be sufficient to determine the qualitative nature of the process gas stream. This data should include the average fractional amounts of $CO_2$, CO, $O_2$, $N_2$, and $H_2O$ in the process effluent gas stream during normal operation. Depending on the industrial process under consideration, there may also be measurable amounts of $NH_3$, $SO_2$, $SO_3$, $NO_x$, HF, sulfuric acid mist, or other volatile substances.

Other qualitative information which should be gathered includes the average gas temperature at the baghouse inlet and outlet, and the average actual and standard volumetric flow rates through the structure.

### 2.4.3 Quantitative Gas Analysis

Flue gas constituents normally specified for analysis are $N_2$, $O_2$, CO, and $CO_2$, and $H_2O$. In addition to these analyses, $SO_2$ and $SO_3$ concentrations are usually measured and sometimes $NH_3$, $NO_x$, HF, or other vapor concentrations are determined.

Oxygen, CO, and $CO_2$ concentrations are measured with a commercial Orsat-type apparatus. Two Orsat-type analyzers are used to determine the oxygen content of the gas entering and leaving the baghouse simultaneously. Comparisons of the inlet and outlet oxygen concentration provides a check for leakage of gas into or out of the baghouse. Although in principle leakage can be determined from an examination of inlet and outlet gas velocity profiles, in practice, flow disturbances at available sampling locations often severely limit the accuracy with which flow determination can be made. Therefore, the simultaneous inlet and outlet oxygen determinations may be a more sensitive indicator of the physical integrity of the baghouse casing.

The Environmental Protection Agency has developed several Stationary Source Test Methods for the determination of various flue gas components. These methods, of course, are not the only way by which the quantity of the gases can be measured. There are many other acceptable analytical methods developed by different testing societies such as the ASME and ASTM. EPA Test Method Number 4 (Federal Register, 1977) describes a procedure to determine the $H_2O$ content of the flue gas. EPA Test Method Number 6 (Federal Register, 1977) can be used to determine the $SO_2$ content of the flue gas. EPA Test Method Number 7 (Federal Register, 1977) explains a method for measuring the nitrogen oxide in the flue gas. The amount of sulfuric acid mist and $SO_2$ content can be determined using the EPA Test Method Number 8 (Federal Register, 1977) (see Appendix E).

Figure 39 illustrates a system not described in the Federal Register, which has been found to be accurate and convenient to use for measurements of $SO_2$ and $SO_3$ concentrations. This is the Controlled Condensation System developed for the EPA by TRW Systems Group (Maddalone and Garner, 1977) to accurately determine $SO_2$ and $SO_3$ concentrations in process gas streams. This procedure

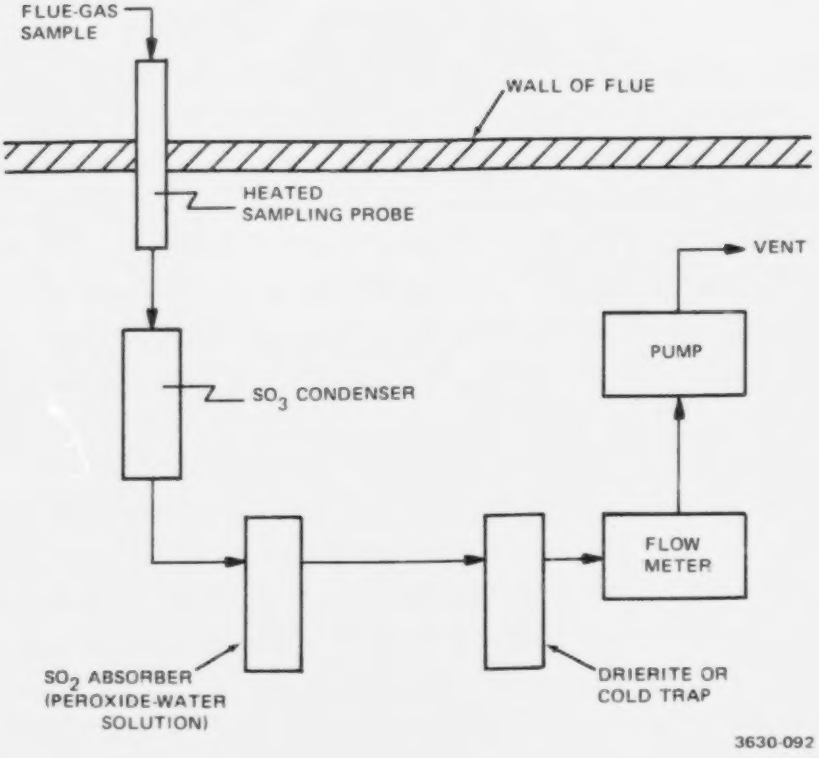

FLUE-GAS SAMPLE
WALL OF FLUE
HEATED SAMPLING PROBE
VENT
PUMP
$SO_3$ CONDENSER
FLOW METER
$SO_2$ ABSORBER (PEROXIDE-WATER SOLUTION)
DRIERITE OR COLD TRAP
3630-092

*Figure 38.* *Schematic diagram of apparatus for the collection of $SO_3$ by the condensation method.*

is applicable in high or low mass loading environments with temperatures up to 300°C and $SO_2$ concentrations up to 600 ppm.

The Controlled Condensation System is based on the separation of $SO_3$ as $H_2SO_4$ from $SO_2$ by cooling the gas stream below the dewpoint of $H_2SO_4$, but above the $H_2O$ dewpoint. Cooling is accomplished by a water-jacketed coil where the $H_2SO_4$ is collected. Particulate matter is collected by a quartz filter mat inserted in the line prior to the condensation coil. The particulate filter system is maintained at a temperature of 288°C to insure that none of the $H_2SO_4$ will condense on the filter mat or filter holder.

The Controlled Condensation Coil (CCC) is a modified Graham Condenser. The water jacket is maintained at 60°C. This is adequate to reduce the flue gas below the dewpoint of the $H_2SO_4$. Following the CCC are two impingers for removing the $SO_2$ and $H_2O$. The $SO_2$ scrubber is a bubbler filled with a 3% solution of $H_2O_2$ in water. The water vapor is removed by a silica gel filled impinger. A vacuum pump with a capacity of 472 $cm^3$/sec is recommended. The total volume of gas sampled is measured with a dry gas meter.

The sampling rate/time is normally 135 $cm^3$/sec for one hour. An indication of the proper amount of sample comes from watching the condenser coil. When the $H_2SO_4$ fog has crept about one-half to two-thirds of the way along the coil, the sampling can cease. After sampling has been completed, the probe and coil are rinsed with deionized $H_2O$ and the recovered solution is analysed in the lab. The amount of $H_2SO_4$ in the condensation coil and probe can be determined by a sulfate or $H^+$ titration. Because of its simplicity and sensitivity, the $H^+$ titration is preferred. This recommended acid/base titration uses Brom phenol Blue as the indicator, since the endpoint of the NaOH and $H_2SO_4$ titration falls near the pH range (3-4.6) of the Brom phenol Blue color change. Complete details can be found in the reference above.

# 3. DEVELOPMENT OF TEST PLANS FOR FABRIC FILTER EVALUATIONS

## 3.1 OBJECTIVES OF CONTROL DEVICE TESTS

Several reasons exist for performing fabric filter control device evaluations. These reasons may range from a verification of compliance with emissions requirements, to programs related strictly to research.

The majority of stationary air pollution sources need some type of control device to satisfy the national, state, or local air pollution regulations that limit the allowable emissions. In order to determine whether the plant is in compliance with these regulations, tests are performed to measure the amount of air pollutant emissions from the control device in question. This is one type of control device evaluation and it is usually the simplest and least expensive.

Another reason for performing tests on a fabric filter control device is to optimize the performance of the installation. These tests might be requested by the owners of the plant where the control device is installed, or by the control device manufacturer. Usually tests of both the inlet and outlet particulate mass concentration are made resulting in a measure of the particulate collection efficiency. In some instances the fractional efficiency (efficiency as a function of particle size) is desired and measurements of the particle size distributions of the inlet and outlet dusts are necessary.

If a particular fabric filter installation is performing poorly due to poor maintenance, or poor design, etc., then tests might be required in order to obtain data to be used in designing additional or replacement fabric filter units.

To obtain data for purely research purposes is a fourth reason for performing a fabric filter control device evaluation. In each test the data may be used to confirm existing theories of fabric filter operation or to develop new theories for modelling and predicting baghouse performance. Research tests may involve total systems studies on the source/control device combination. These tests are usually the most complicated and expensive because of the amount of data that is desired.

## 3.2 TYPE AND NUMBER OF TESTS REQUIRED

As mentioned in the previous section, the type and number of tests that are performed during a fabric filter evaluation depend on the reason for the tests and the amount of funding available.

In most all cases the standard compliance test involves a determination of the particulate mass concentration at the control device outlet. Depending on the type of source, some measurements of the gaseous emissions may also be required. The minimum number of tests to be performed during a compliance test is usually set by Federal or State regulations.

In order to study the performance of a fabric filter, measurements of both the inlet and outlet mass concentration are performed. These data are required for calculations of the particulate collection efficiency. If the collection efficiency is to be related to particle size, particle size measurements must be performed at the inlet and outlet. If the source is stable, fewer tests will be required than if the plant process is cyclic or variable over an indeterminant time period. If a fabric filter appears to be performing poorly, then other tests might be necessary depending on the type of problem encountered. At a fabric filter installation, the problem might be torn bags in one baghouse compartment. This might require a special test strategy to isolate this compartment.

Data that are required for fabric filter design are the particulate mass concentration; the particle size distribution; the physical and chemical proeprties of the dust to be collected; and the effluent gas temperature, pressure, and composition. A fairly extensive testing program is necessary in order to obtain these data. Tests should be performed during all normal process cycles and with all types of expected feedstock to insure that the fabric filter will not be designed undersize.

If testing is to be performed on a fabric filter for research purposes only, then the tests that are made are dependent on the information which is desired as well as the amount of funding. As is true of all experimental type programs, the more data that are obtained, the more reliable will be the conclusions based on those data. Usually control device research programs are designed to gather as much information as practical for the money available. Generally, research studies concern the particulate mass concentrations at the inlet and outlet, the inlet and outlet particle size distributions, gas analysis, the dust properties, the control device operation parameters, plant process data, previous control device maintenance data, and the economics of the particular control device. Of course, the type of fabric filter and plant will determine the specific tests which are conducted.

In some instances the type of tests which are conducted depend on cooperation from the plant personnel. They may or may not be willing to alter the feedstock or change the settings on the particle collector controls, for example. Usually these problems are worked out as the test plan is developed.

In summary, fabric filter testing is not a routine operation that has had all the problems worked out or specific procedures developed. Each plant-control device combination is unique and should be treated as such. Certain problems exist at one installation which might not be encountered at any other control device installation. The number of specific tests which should be performed will depend on the type of control device, the stability of the source, the length of time allowed for testing, and the available funding. It is usually advisable to perform as many tests as practical to insure that adequate data exist, because later it may be found that some tests must be disqualified.

Table IV indicates some of the considerations and problems that must be dealt with in developing a test plan for general control device evaluations. Although this table is designed to serve as a planning outline, the relative importance of the facets of the plan, or considerations that are not listed, can only be established from a good understanding of the plant-control device system and the objectives of the test.

For the purposes of developing specific baghouse test plans, three basic levels of source-fabric filter data can be identified. Table V depicts these categories in a flow chart where the progression is from minimum data to maximum data, or from relatively inexpensive and qualitative to very expensive and quantitative programs. In general, a basic test plan can be developed around this flow chart. Depending on the interest in different types of information, as well as the amount of funding available, the actual test plan would be modelled after a Level A, B, or C fabric filter evaluation.

In the following sections each of the components of these test plans are briefly discussed. Also, a list of important items to be noted under each category is presented.

# TABLE IV

## PARTICULATE CONTROL DEVICE TASKS

| Objective of Tests | Assure Compliance with EPA Regulation | Optimize Performance of Control Device | Obtain Design Data for Control Device | Obtain Data for Modeling Studies | Systems Studies Process and Control Device |
|---|---|---|---|---|---|
| Tests Required | | | | | |
| Mass Concentration | O | O | I | I,O | I,O |
| Opacity | O | O | | | O |
| Gas Composition | O | O | I | I,O | I,O |
| Gas Temperature | | | I | I,O | I,O |
| Gas Volume | O | | I | I,O | I,O |
| Pressure | | | I | I,O | I,O |
| Velocity Distribution | | C | C | X | X |
| Particle Size Distribution | | | I | I,O | I,O |
| Dust Composition | | | Qualitative | Qualitative | I,O* |
| Dust Resistivity | | | | ESP only | X |
| Control Device Data | | | | | |
| Operating | | | | X | X |
| Design | | | | X | X |
| Maintenance | | | | | X |
| Plant Process Data | | | | D | D |
| Economic Data | | | | Optional | X |
| Technical Considerations (Decisions/Problems) | | | | | |
| Adequate Space, Electric Power | O | O | I | I,O | I,O |
| Laboratory Space | X | X | X | X | X |
| Number of Tests Required | 3 | D | D | D | D |
| Isokinetic Sampling | X | X | X | X | X |
| Condensible Vapors/ Volatile Particles | | C | C | C | C |
| Mass Concentration/ Sampling Time | C | C | C | C | C |
| Traverse Strategy | O | O | O | O | O |
| Aerosol Gas Velocity | C | C | C | C | C |
| Process/Emission Variations | C | C | C | C | C |
| Select Particle Sizing Methods | | | I,O | I,O | I,O |
| Select Mass Train Type | O | O | I,O | I,O | I,O |
| Select Gas Analysis Methods | | D | D | D | D |
| Re. Time Monitors Needed | | D | D | D | D |
| Filter Mass Stability | | C | C | C | C |
| Sample Preservation | | D | D | D | D |

Key:
O · Outlet
I · Inlet
X · Required
D · Decision based on specific site or test objectives
C · Must be considered
* vs. Particle Diameter

## TABLE V

## THREE LEVELS OF EFFORT FOR FABRIC FILTER EVALUATION

| LEVEL | | | |
|---|---|---|---|
| C | B | A | |
| ↓ | ↓ | ↓ | ENGINEERING AND OPERATING DATA<br>Baghouse Design, Operating and Maintenance Data<br>Qualitative Gas Composition<br>Compartment Pressure Drop Data<br>Opacity Monitor Check For Leaks<br>Operating Air-to-Cloth Ratio<br>Two men on site for two days. Two man weeks for analysis and reporting. |
| ↓ | ↓ | | PARTICULATE MASS CONCENTRATION AND FABRIC FILTER EFFICIENCY<br>Pretest Site Survey, Port Installation<br>Inlet and Outlet Mass Sampling<br>Quantitative Gas Analysis<br>Five men on site for five days. One man month for analysis and reporting. |
| ↓ | | | PARTICLE SIZING AND FABRIC FILTER FRACTIONAL EFFICIENCY<br>Inlet and Outlet Particle Size Distribution<br>Ten to fifteen men on site for one or two weeks<br>Six to twenty man months for analysis, modelling, and reporting. |

### 3.2.1 Fabric Filter Level A Evaluation

A Level A Fabric Filter Evaluation includes baghouse design, operating, and maintenance data, baghouse compartment pressure drop data, qualitative flue gas analysis, operating air-to-cloth ratios, and opacity monitor information for each baghouse compartment.

#### 3.2.1.1 Plant Operating Data

Generally the main inputs in plant operating data are the raw input materials, the output materials, and the waste effluent, as well as the conditions of plant operation. Sometimes the material collected by the fabric filter is the prime plant product or is a by-product which is recycled or sold. An important aspect of plant operation data is the variability of the source. The plant may have periods of high or low output, or it may be cyclic over a predetermined period. Also the raw materials may vary in composition over this cycle. The following list encompasses most areas which should be noted. This data is normally available from plant personnel.

a. Raw input materials
b. Output products
c. Waste effluent materials
d. Qualitative waste gas composition
e. Qualitative waste particulate effluent
f. Normal plant operating cycles
g. Normal plant outage periods
h. Plant economics: unit input energy/unit output product
i. Plant operating peculiarities

### 3.2.1.2 Baghouse-Fabric Filter Design Data

All aspects of baghouse-fabric filter design should be noted, if available. This includes structural design and filter design data. This information will usually be available from plant personnel and manufacturer's literature.

a. Type of baghouse - number of baghouses, manufacturer
b. Dimensions of baghouse
c. Inlet and outlet manifolds
d. Number of baghouse compartments
e. Fans and capacity
f. Compartment accessibility
g. Number of bags
h. Number of bags per compartment
i. Bag shape and size
j. Bag construction
k. Bag placement and spacing
l. Bag cleaning mechanism
m. Pressure drop to initiate cleaning
n. Bag cleaning cycles
o. Type of fabric filter material
p. Fiber and fabric characteristics
q. Fabric treatments
r. Fabric thickness and weight
s. Air-to-cloth ratio
t. Dust removal systems

Refer to Sections 1.1, 2.1, and 2.2.

### 3.2.1.3 Flue Gas Characteristics, Baghouse $\Delta P$, Maintenance Data

Under this section actual quantitative data concerning baghouse operation is noted. This includes gas flow rates, temperature, pressure drops during filtering and cleaning cycles across each baghouse compartment, and any maintenance data that would be useful in evaluating the performance of the baghouse. Maintenance data might include bag failure data, bag replacement scheduling, start-up difficulties, etc. A sample check list for noting potential problems is presented in Table VI.

a. Effluent gas volumetric flow rates
b. Qualitative gas composition ($H_2O$, $O_2$, $N_2$, $CO_2$, CO)
c. Qualitative evidence of $SO_2$, $SO_3$, $H_2SO_4$ and other gas forms depending on plant process
d. Gas temperatures
e. Pressure drop cycles across each compartment
f. Bag failures, opacity monitor data for each compartment
g. Bag replacement schedule
h. Any start-up difficulties
i. Current bag conditions
j. General maintenance status

Refer to Sections 2.1.2 and 2.1.5.

TABLE VI

MAINTENANCE REPORT - TYPE AND FREQUENCY OF PROBLEMS

| Problem Type | Frequency |
|---|---|
| 1. Fabric - dust deposit interactions | |
| a. intestitial deposit related, abrasion, wear | |
| b. flexture wear failures | |
| c. seeping | |
| d. blinding | |
| e. burning, heat | |
| f. holes, pinholes, shot holes | |
| g. hygroscopicity | |
| h. condensation | |
| i. hardened cake tears, cracked bags | |
| j. others | |
| 2. Fabrication failures, mechanical bag failures | |
| a. bag seams, sewing failures | |
| b. bag top tears | |
| c. others | |
| 3. Design or maintenance failures related to tensioning, supports, rings, collars, or cleaning device interactions. | |
| a. chafing on housing or other bags | |
| b. tensioning, bags too loose | |
| c. cage, wire, ring abrasion wear, support mechanism interaction | |
| d. cleaning carriage bag wear | |
| e. seals around cloth-metal collars | |
| f. others | |

TABLE VI (Continued)

| Problem Type | Frequency |
|---|---|
| 4. Collector design problems; internal, external mechanisms | |
| a. unable to enter collector to service or maintain during operation | |
| b. hole detection problems | |
| c. hopper dust sticking, holdup, screw conveyor plugging. | |
| d. internal mechanism wear | |
| e. external mechanism wear, timer, shaker, fan, bearings, doors, seals, wall failures, etc. | |
| f. others | |
| 5. Dust collecting system design problems, external to collectors (inc. piping, hoods) | |
| a. piping, elbows, abrasion | |
| b. hood inlet control poor | |
| c. others | |

### 3.2.2. Fabric Filter Level B Evaluation

A Level B Fabric Filter Evaluation includes all elements of a Level A Evaluation as well as Quantitative Gas Analysis and Inlet and Outlet Mass Concentration Measurements. These concentration measurements will allow a calculation of the Total Mass Collection Efficiency.

#### 3.2.2.1 Quantitative Gas Analysis

Quantitative gas analysis involves actual measurements on the effluent gases to determine the components of the gas mixture. This usually includes at the minimum determinations of the water vapor content and amounts of $N_2$, $O_2$, $CO_2$, and CO. Depending on the type of plant, determinations of the $SO_2$ and $SO_3$ content will be necessary. At some installations the flue gas or filter bags are treated with Nahcolite or other $SO_2$ adsorbing materials to remove this gas. In these cases measurements of the $SO_2$ content upstream and downstream of the baghouse might be required. Also depending on the particular industrial process, there may be other specific gas components which might affect baghouse operation.

a. ORSAT Measurement of $O_2$, $N_2$, CO, and $CO_2$
b. EPA Reference Methods for $H_2O$, $SO_2$, $H_2SO_4$, $NO_x$
c. Alternate methods for determining $SO_2$ and $SO_3$
d. Other gases peculiar to particular site
e. Inlet and outlet $SO_2$, if $SO_2$ removal chemicals being injected.

Refer to Section 2.5.3.

### 3.2.2.2 Inlet and Outlet Mass Concentration Measurements Total Mass Collection Efficiency

In order to determine the overall mass collection efficiency, the mass concentration of the effluent particulate at the inlet and outlet of the baghouse must be determined. Usually this will involve either the EPA Reference Method Number 5 or the ASTM method as described under Section 2. In most cases simultaneous inlet and outlet sampling will be necessary, as well as a traversing capability. Also sampling times at the inlet and outlet will be very different due to the usual excellent efficiency of baghouse units.

a. Sampling train type
b. Flue depth
c. Traversing capability
d. Velocity/Temperature Traverse
e. Probe heating required
f. Filter integrity
g. Isokinetic sampling
h. Sampling times
i. Number of samples
j. Process variations
k. Integrated or time averaged sample

Refer to Appendix B and Appendix E.

### 3.2.3 Fabric Filter Level C Evaluation

A Level C Fabric Filter Evaluation includes the data gathered under a Level B Evaluation and Inlet and Outlet Particle Size Distribution Measurements. This data will allow determination of the control device fractional efficiency.

To measure the collection efficiency of a baghouse as a function of particle size or the fractional efficiency, determinations of the inlet and outlet particle size distributions must be made. There are several types of instruments which can be used for this purpose. These include cascade impactors, series cyclones, optical particle counters, diffusion batteries/condensation nuclei counters, and electrical aerosol analyzers. In general process variations will have an effect on sampling times for impactors and cyclones at baghouse inlets. Recent experimental data show that outlet emissions from seasoned baghouses are fairly stable even if the inlet concentration is fluctuating. Thus inlet sampling devices may have to sample during many parts of a cyclic operation to obtain a representative inlet size distribution.

Several sizing systems are listed below along with several aspects of operation to be considered in developing a test plan.

Cascade Impactors

| | |
|---|---|
| Size Range | 0.3-10.0 micrometers |
| a. High Loading | Low Loading |
| Brink | Andersen |
| | MRI |
| | Sierra |
| | U. of W. |

b. Isokinetic Sampling
c. Jet Velocity Limits
d. Nozzle Selection
e. Precutter Selection
f. Loading Limits

Refer to Appendix C and Section 2.3.2.

### Series Cyclones

| | |
|---|---|
| Size Range | 0.3-10 micrometers |

a. Accuracy/Resolution
b. Sampling Times
c. Back Up Filters
d. Filter Integrity
e. Filter Loading
f. Constant Flow Rate
g. Traversing
h. Isokinetic Sampling
i. Extractive Sampling
j. Probe Losses

Refer to Section 2.3.2.

### Optical Particle Counters

| | |
|---|---|
| Size Range | 0.3-10 micrometers |

Refer to Section 2.3.3.

### Diffusional/Electrical Aerosol Analyzer

| | |
|---|---|
| Size Range | 0.01-0.3 micrometers |

a. Extractive Sampling
b. Sample Conditioning
c. Dilution of Sample
d. Gas Composition
e. Condensible Vapors
f. Heated Lines
g. Probe Losses
h. Real Time Monitoring
i. Duct Pressures

Refer to Appendix D and Section 2.3.4.

## 3.3 GENERAL PROBLEMS AND CONSIDERATIONS

It is rare that a fabric filter evaluation program does not encounter several problems in performing the required tests. These problems can cover a wide range of circumstances and affect the ability to complete the test program successfully. Although it is impossible to anticipate every contingency, careful planning can reduce the likelihood of complete failure of the test program. A discussion of the more commonly encountered problems and situations is presented below.

### Plant Location

Plant location will generally not be a problem unless it is a long distance to a city where acceptable accommodations and supplies are available, or if the nearest airport is not convenient for shipping equipment or for transportation of personnel. Also, depending on the time of year, the local weather can force postponement of testing, unusual working hours, or require the construction of special shelters for test crew members required to work out of doors.

### Sampling Location and Accessibility

In most new plants the requirements of compliance testing at the control device outlet (ports, platforms, power, etc.) have been taken into account in designing the facility. This is frequently not true of older plants. At many sites, the equipment must be hand carried or hoisted to the sampling location. Stack testing can be difficult without a properly designed platform. Sometimes platforms and scaffolding must be erected to allow direct access to the sampling location. It is recommended that a pre-test site survey be conducted to determine if any platforms or shelter must be built prior to actual testing. This is also a good time to inspect the entire plant and establish contact with the plant employee who will be responsible for liaison with the plant managers.

### Type of Ports

Almost all sampling of flue gases and dusts requires some type of port. Before the tests begin it is advisable to know the location, type, number, and size of the ports that are available (inlet, outlet, stack). For some types of test equipment, the ports may be too small and require replacement with larger diameter ports. The number of ports will also determine the flexibility that one has in planning for traverses of the duct to obtain representative samples. There may also be some difficulties with the type of ports that are installed, whether flanged or threaded internally or externally. Other problems commonly encountered with sampling ports are the length of the port nipple, rusting of port caps onto the nipples, and caking of dust inside the ports which must be chiseled away before sampling can begin, etc.

### Dust Size

The duct size will generally determine whether traversing is feasible. Traversing twenty foot deep ducts is not a simple matter, especially if the probes must be heat traced. Special hoists sometimes must be erected. Small circular ducts usually cannot be effectively traversed, and in some cases, instruments that are normally operated *in situ*, must be operated in an oven with special sampling probes for extracting the samples.

### Flue Gas Velocity and Nozzle Sizes

Depending on the location of the sampling ports, the flue gas velocity can sometimes be very high or very low. Isokinetic sampling is highly desirable when sampling dusts. Gas velocities are usually lowest in the transforms immediately upstream or downstream from control devices. In these instances the nozzle sizes required for isokinetic sampling may be larger than standard sizes. On the other hand high gas velocities can require impractically small nozzles, especially when sampling less than 14 liters per minute. If the concern is with particles smaller than about five micrometers diameter, errors from non-isokinetic sampling are less significant.

## Power Requirements

Depending on the amount of equipment operating at one time, the accessible power outlets at most sampling locations may or may not be adequate. In many instances long extension cords are necessary, and in some cases a transformer is needed to change the available power to 110 volts. Before testing, the power requirements should be calculated and plant personnel contacted if it appears that additional power outlets will be required.

## Gas Temperature and Dew Point

Under some circumstances the gas temperature can cause difficulties. Too high a temperature can cause galling, metal fatigue, collection substrate problems, and poor vacuum sealing for *in situ* sampling equipment. Probes and other sampling equipment may have to be insulated or heat traced to prevent premature cooling of the gases. Low gas temperatures can be especially troublesome when the slightest temperature drop can cause excursion through dew points causing condensation within the probe or on collection filters. $H_2SO_4$ condensation, or chemical reaction can mask particulate weight gains on glass fiber collection substrates. Usually heating to 17°C above the gas dew point is recommended to avoid condensation. At some plant temperature fluctuations can occur as a result of process variations or the amount of excess air in boiler operations. A knowledge of this type of activity is desirable before testing.

## Corrosive Gases

At some locations, particle growth, such as $H_2SO_4$ mists, can mask the true concentration of fine particulate matter. This can only be alleviated by keeping the gas temperature in the sampling train sufficiently high or by dilution with clean dry air. Reevaporation of $H_2SO_4$ mists requires very high temperatures, and this problem can usually more easily be avoided than corrected.

## Volatile Components

In planning an effective sampling protocol, it is necessary to consider whether or not volatile components make up a significant part of the emissions. Smelting processes are a notable example of sources where much of the mass emissions consist of compounds that exist in vapor form at flue gas temperatures, but condense to form solid particles upon cooling in the atmosphere. For process streams such as these, the nature or quantity of the sample is dependent on the temperature of the sampling train. It is usually advantageous to design a special train with several stages kept at progressively lower temperatures, in order to fully understand the nature of the emissions. Sulfuric acid is a good example of a volatile pollutant that can present control and sampling problems.

## Long and Short Sampling Times

In general, control devices are very efficient particle collectors. At the inlet, high dust concentrations may necessitate undesirably short sampling durations. Extremely short (less than five minutes) sampling durations do not allow an adequate period of integration over the plant process cycles unless the cycles are very stable and long. On the other hand, low dust concentrations at control device outlets sometimes require sampling time as long as 12 hours, hampering the study of emissions from each part of a process cycle. It is helpful to use low flow rate sampling devices at control device inlets and high flow rate devices at outlets in order to obtain reasonable sampling times.

## Laboratory Space

Usually arrangements can be made to obtain the use of a portion of the chemical laboratory normally found at most industrial plants. In some cases, however, the location of this laboratory may not be convenient to the sampling site. As part of the pretest site survey, a decision should be made as to whether some type of temporary, mobile lab or trailer would be more convenient than the plant laboratory space.

## Process Cycles and Feedstock Variations

In many plants, such as iron and steel mills and smelting operations, the effluent gas and dust characteristics vary dramatically over a single process cycle. If the test objective is to obtain a good average of the emissions, the sampling time is quite flexible. However, if the test objective is to isolate emissions from a particular part or from each part of an average cycle, the sampling time must be short, or the tests interrupted periodically and run only during the part of interest. At some plants the supply of fuel or feedstock can change. Normally a plant will maintain logs of the important process parameters, and this information should be obtained and correlated with the test data. This can avoid costly repetition of test procedures or invalidation of the test data.

# APPENDIX A
# AEROSOL FUNDAMENTALS, NOMENCLATURE, AND DEFINITIONS

## A.1 GENERAL DISCUSSION

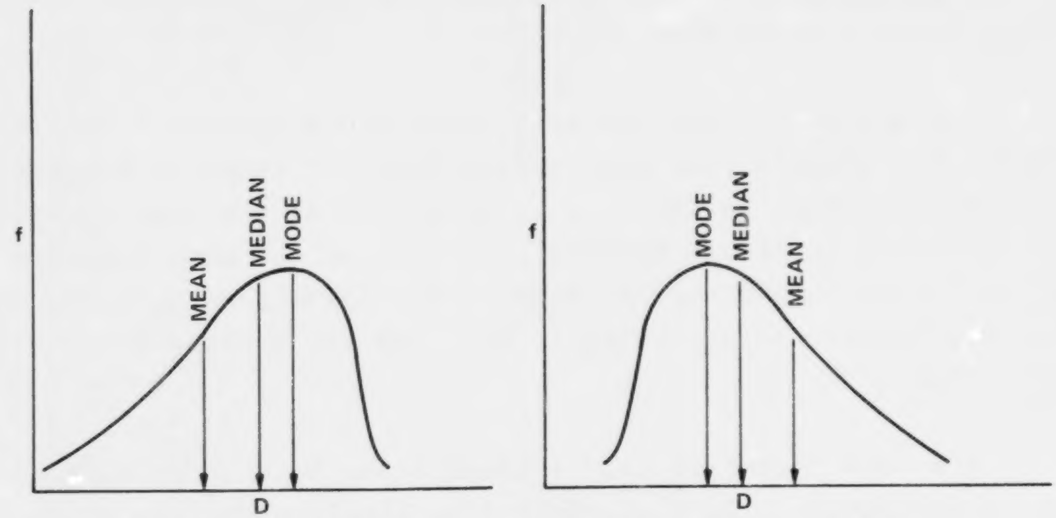
The particulate matter suspended in industrial gas streams may be in the form of nearly perfect spheres, regular crystalline forms other than spheres, irregular or random shapes, or as agglomerates made up from combinations of these. It is possible to discuss particle size in terms of the volume, surface area, projected area, projected perimeter, linear dimensions, light scattering properties, or in terms of drag forces in a liquid or gas (mobility). Particle sizing work is frequently done on a statistical basis where large numbers of particles, rather than individuals, are sampled. For this reason the particles are normally assumed to be spherical. This convention also makes transformation from one basis to another more convenient.

Experimental measurements of particle size normally cannot be made with a single instrument if the size range of interest extends over much more than a decimal order of magnitude. Presentations of size distributions covering broad ranges of sizes then must include data points which may have been obtained using different physical mechanisms. Normally the data points are converted by calculation to the same basis and put into tabular form or fitted with a histogram or smooth curve to represent the particle size distribution. Frequently used bases for particle size distributions are the relative number, volume, surface area, or mass of particles within a size range. The size range might be specified in terms of aerodynamic, Stokes, or equivalent PSL diameter. There is no standard equation for statistical distributions which can be universally applied to describe the results given by experimental particle size measurements. However, the log-normal distribution function has been found to be a fair approximation for some sources of particulate matter and has several features which make it convenient to use. For industrial sources the best procedure is to plot the distribution in different size ranges separately, rather than trying to characterize the entire distribution by two or three parameters. The ready availability of inexpensive programmable calculators which can be used to convert from one basis to another compensates greatly for the lack of an analytical expression for the size distribution.

## A.2 PARTICLE SIZE DISTRIBUTIONS

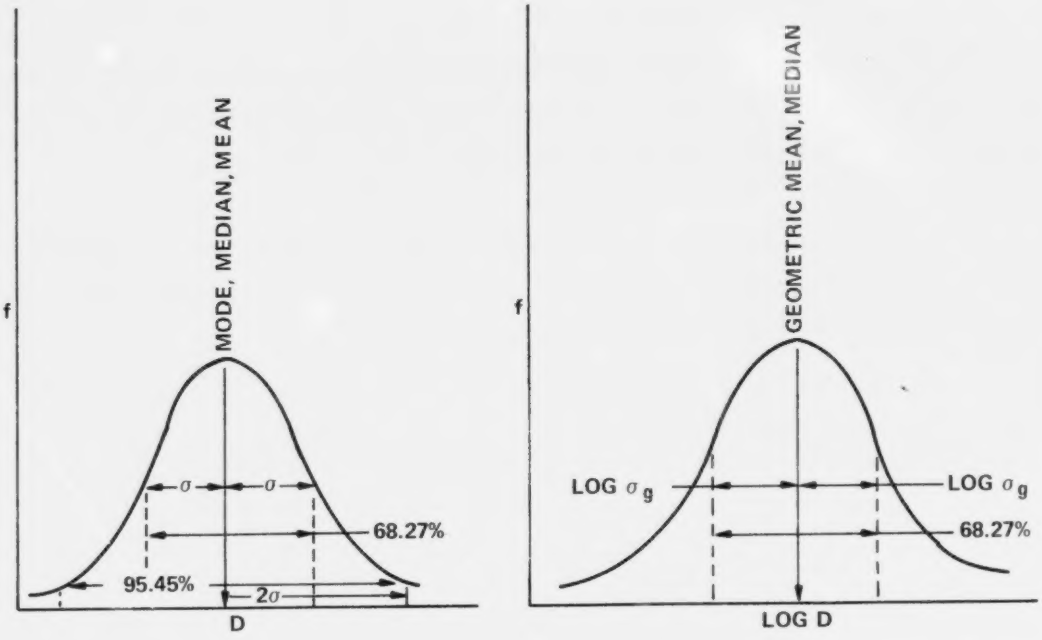
Figure A1 shows plots of generalized unimodal particle size distributions which will be used to graphically illustrate the terms which are commonly used to characterize an aerosol. Occasionally size distribution plots exhibit more than one peak. A size distribution with two peaks would be called bimodal. Such distributions can frequently be shown to be equivalent to the sum of two or more distributions of the types shown in Figure A1. If a distribution is symmetric or bell shaped when plotted along a linear abscissa, it is called a "normal" distribution (Figure A1c). A distribution that is symmetric or bell shaped when plotted on a logarithmic abscissa is called "log-normal" (Figure A1d).

Interpretation of the frequency or relative frequency shown as f in Figure A1 is very subtle. One is tempted to interpret this as the amount of particulate of a given size. This interpretation is erroneous, however, and would require that an infinite number of particles be present. The most useful convention is to define f in such a way that the area bounded by the curve



a. Distribution Skewed Left

b. Distribution Skewed Right



c. Normal Distribution

d. Log Normal Distribution

3630-091

*Figure A1. Examples of frequency or particle size distributions. D is the particle diameter.*

(f) and vertical lines intersecting the abscissa at any two diameters is equal to the amount of particulate matter in the size range indicated by the diameters selected. f then is equal to the relative amount of particulate matter in a narrow size range about a given diameter.

The median divides the area under the frequency curve in half. For example, the mass median diameter (MMD) of a particle size distribution is the size at which 50% of the mass consists of particles of larger diameter, and 50% of the mass consists of particles having smaller diameters. Similar definitions apply for the number median diameter (NMD) and the surface median diameter (SMD).

The term "mean" is used to denote the arithmetic mean of the distribution. In a particle size distribution the mass mean diameter is the diameter of a particle which has the average mass for the entire particle distribution. Again, similar definitions hold for the surface and number mean diameters.

The mode represents the diameter which occurs most commonly in a particle size distribution. The mode is seldom used as a descriptive term in aerosol physics.

The geometric mean diameter is the diameter of a particle which has the logarithmic mean for the size distribution. This can be expressed mathematically as:

$$\log D_g = \frac{\log D_1 + \log D_2 + \ldots . \log D_N}{N} \qquad \text{(A1a)}$$

or as

$$D_g = \left(D_1 D_2 D_3 \; . \; . \; . \; . \; D_N\right)^{1/N} \tag{A1b}$$

The standard deviation ($\sigma$) and relative standard deviation ($\alpha$) are measures of the dispersion (spread, or polydispersity) of a set of numbers. The relative standard deviation is the standard deviation of a distribution divided by the mean, where $\sigma$ and the mean are calculated on the same basis; i.e., number, mass, or surface area. A monodisperse aerosol has a standard deviation and relative standard deviation of zero. For many purposes the standard deviation is preferred because it has the same dimensions (units) as the set of interest. In the case of a normal distribution, 68.27% of the events fall within one standard deviation of the mean, 95.45% within two standard deviations, and 99.73% within three standard deviations.

### A.2.1 Cumulative and Differential Graphs

Field measurements of particle size usually yield a set of discrete data points which must be manipulated or transformed to some extent before interpretation. The resultant particle size distribution may be shown as tables, histograms, or graphs. Graphical presentations are the conventional and most convenient format and these can be of several forms.

Cumulative size distributions -- Cumulative mass size distributions are formed by summing all the mass containing particles less than a certain diameter and plotting this mass versus that diameter. The ordinate is specifically equal to

$$M(j) = \sum_{t=1}^{j} M_t.$$

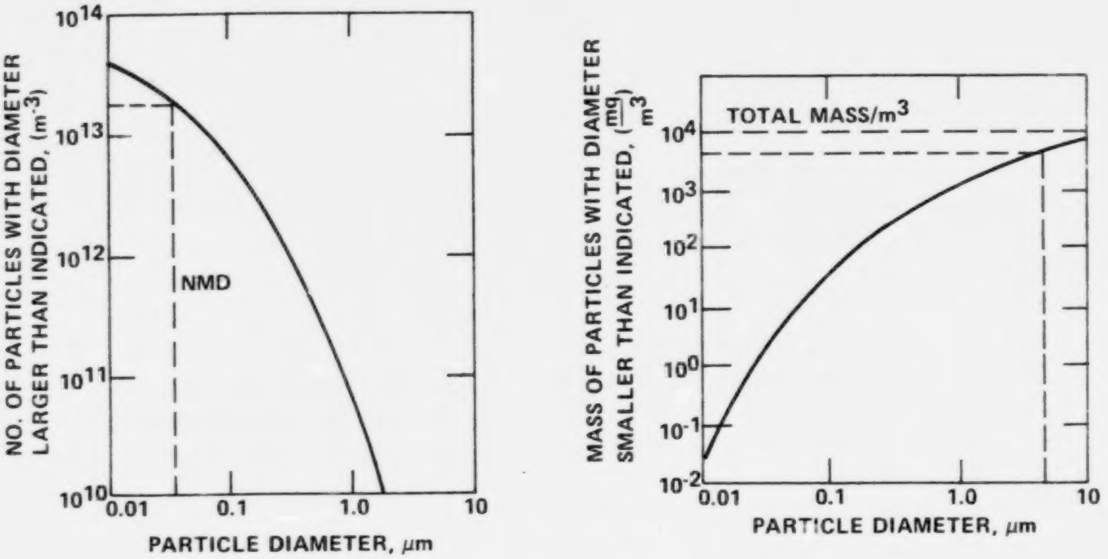
where $M_t$ is the amount of mass contained in the size interval between $D_j$ and $D_{j-1}$.

The abscissa would be equal to $D_j$. Cumulative plots can be made for surface area and number of particles per unit volume in the same manner. Examples of cumulative mass and number graphs are shown in Figures A2b and A2a, respectively, for the effluent from a coal fired power boiler. Although cumulative plots obscure some information, the median diameter and total mass per unit volume can be obtained readily from the curve. Because both the ordinate and abscissa extend over several orders of magnitude, logarithmic axes are normally used for both.

A second form of cumulative plot which is frequently used is the cumulative percent of mass, number, or surface area contained in particles having diameter smaller than a given size. In this case the ordinate would be, on a mass basis:

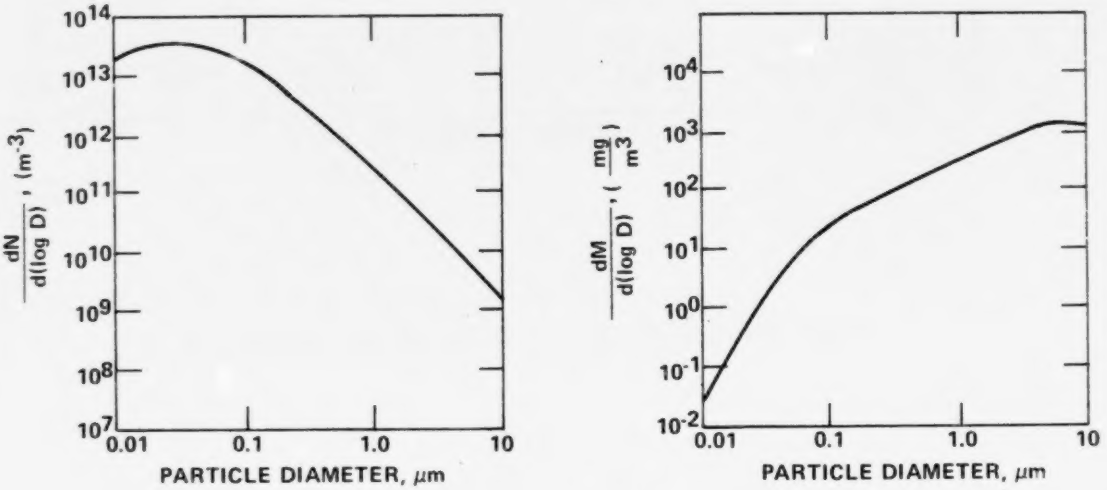
$$\text{Cumulative percent of mass less than size} = \frac{\sum_{t=1}^{j} M_t}{\sum_{t=1}^{N} M_t} \times 100\%. \qquad \text{(A2)}$$

The abscissa would be log $D_j$. Special log-probability paper is used for these graphs, and for log-normal distributions the data set would lie along a straight line. For such distributions the median diameter and geometric standard deviation can be easily obtained graphically. Figures A3a and A3b show cumulative percent graphs for the size distribution shown in Figure A2a and a log-normal size distribution.



a. **Cumulative No. Graph**

b. **Cumulative Mass Graph**

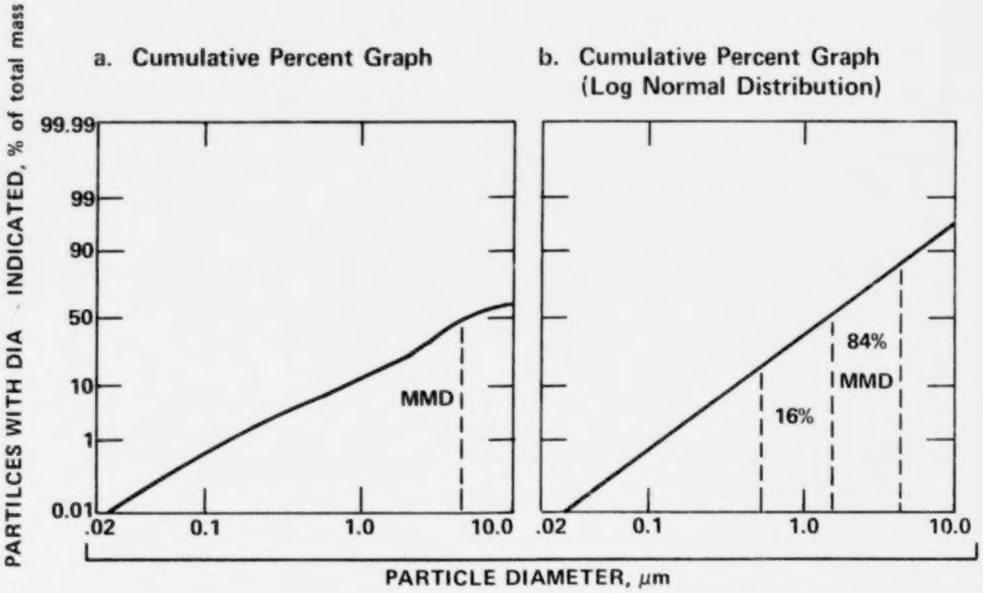


c. **Differential No. Graph**

d. **Differential Mass Graph**

3630-089

*Figure A2. A single particle size distribution presented in four ways. The measurements were made in the effluent from a coal-fired power boiler.*



3630-087

*Figure A3. Size distributions plotted on log probability paper.*

Differential size distributions -- Differential particle size distribution curves are obtained from cumulative plots by taking the average slope over a small size range as the ordinate and the geometric mean diameter of the range as the abscissa. If the cumulative plot were made on logarithmic paper the frequency (slope) would be, taking finite differences:

$$\frac{\Delta M}{\Delta(\log D)} = \frac{M_j - M_{j-1}}{\log D_j - \log D_{j-1}}, \qquad \text{(A3)}$$

and the abscissa would be $D_g = \sqrt{D_j D_{j-1}}$ where the size range of interest is bounded by $D_j$ and $D_{j-1}$. $M_j$ and $M_{j-1}$ correspond to the cumulative masses below these sizes. Differential number and surface area distributions can be obtained from cumulative graphs in precisely the same way. Differential graphs show visually the size range where the particles are concentrated with respect to the parameter of interest. The area under the curve in any size range is equal to the amount of mass (number, or surface area) consisting of particles in that range, and the total area under the curve corresponds to the entire mass (number, or surface area) of particulate in a unit volume. Again, because of the extent in particle size and the emphasis on the fine particle fraction, these plots are normally made on logarithmic scales. Figures A2c and A2d are examples of differential graphs of particle size distributions.

Log-normal size distributions -- The formation of aerosols by different means frequently result in particle size distributions which obey the log-normal law. For log-normal particle size distributions the geometric mean and median diameters coincide.

The normal distribution law is, on a mass basis:

$$f = \frac{dM}{dD} = \frac{1}{\sigma\sqrt{2\pi}} \exp\left[-\frac{\left(D-D_m\right)^2}{2\sigma^2}\right]. \tag{A4}$$

The log-normal distribution law is derived from this equation by the transformation D → log D

$$f = \frac{dM}{d(\log D)} = \frac{1}{\log \sigma_g\sqrt{2\pi}} \exp\left[-\tfrac{1}{2}\left(\frac{\log D - \log D_{gm}}{\log \sigma_g}\right)^2\right] \tag{A5}$$

where $\sigma_g$, the geometric standard deviation, is obtained by using the transformation D → log D in equation A4. This distribution is symmetric when plotted along a logarithmic abscissa and has the feature that 68.3% of the distribution lies within one geometric standard deviation of the geometric mean on such a plot. Mathematically, this implies that $\log \sigma_g = \log D_{84.14} - \log D_g$ or $\log D_g - \log D_{15.86}$ where $D_{84.14}$ is the diameter below which 84.14% of the distribution is found, etc. This can be simplified to yield:

$$\sigma_g \simeq \frac{D_{84}}{D_g}, \tag{A6}$$

$$\sigma_g \simeq \frac{D_g}{D_{16}}, \text{ or} \tag{A7}$$

$$\sigma_g \simeq \left(\frac{D_{84}}{D_{16}}\right)^{\frac{1}{2}} \tag{A8}$$

When plotted on log-probability paper, the log-normal distribution is a straight line on any basis and is determined completely by the knowledge of $D_g$ and $\sigma_g$. This is illustrated in Figure A3b. Another important feature is the relatively simple relationships among log-normal distributions of different bases. If $D_{gm}$,

$D_{gs}$, $D_{gvs}$, and $D_{gN}$ are the geometric mean diameter of the mass, surface area, volume-surface, and number distribution, then:

$$\log D_{gs} = \log D_{gm} - 4.6 \log^2 \sigma_g , \tag{A9}$$

$$\log D_{gvs} = \log D_{gm} - 1.151 \log^2 \sigma_g , \text{ and} \tag{A10}$$

$$\log D_{gN} = \log D_{gm} - 6.9 \log^2 \sigma_g . \tag{A11}$$

The geometric standard deviation remains the same for all bases.

More examples of particle size distribution graphs are given in the data reduction sections of Appendices C and D. The following section in this Appendix lists useful definitions, equations, and nomenclature for aerosol sampling.

## A.3 NOMENCLATURE AND DEFINITIONS

### A.3.1 Definition of Particle Diameter

*Aerodynamic diameter, $D_A$* -- The aerodynamic diameter of a particle is the diameter of a sphere of unit density which has the same settling velocity in the gas as the particle of interest.

*Aerodynamic impaction diameter, $D_{AI}$* -- The $D_{AI}$ of a particle is an indication of the way that a particle behaves in an inertial impactor or in a control device where inertial impaction is the primary mechanism for collection. If the particle Stokes diameter is known, $D_s$, the $D_{AI}$ is equal to:

$$D_{AI} = D_s \sqrt{\rho C} , \tag{A12}$$

where $\rho$ is the particle density, gm/cm$^3$, and
C is the slip correction factor.

Stokes diameter, $D_S$ -- If the density of a particle is known, the Stokes diameter may be used to describe particle size. This is the diameter of a sphere having the same density which behaves aerodynamically as the particle of interest. For spherical particles, the Stokes number is equal to the actual dimensions of the particle.

An average density for the particles can be obtained from volume-weight data using a helium pycnometer if large enough samples are available. The validity of size information based on an average density depends upon the uniformity of the density from particle to particle, particularly with respect to size. Visual inspection of some size-classified samples from flue gases sometimes shows a variation in color with size which would seem to indicate compositional inhomogeneities.

Equivalent polystyrene latex (PSL) diameter -- The intensity of light scattered by a particle at any given angle is dependent upon the particle size, shape, and index of refraction. It is impractical to measure each of these parameters and the theory for irregularly shaped particles is not well developed. Sizes based on light scattering by single particles are therefore usually estimated by comparison of the intensity of scattered light from the particle with the intensities due to a series of calibration spheres of very precisely known size. Most commonly these are PSL spheres.* Spinning disc and vibrating orifice aerosol generators can be used to generate monodisperse calibration aerosols of different physical properties. Because most manufacturers of optical particle sizing instruments use PSL spheres to calibrate their instruments, it is convenient to define an equivalent PSL diameter as the diameter of a PSL sphere which gives the same response with a particular optical instrument as the particle of interest.

---

*Available from The Dow Chemical Company, P.O. Box 68511, Indianapolis, Indiana 46268.

Equivalent volume diameter -- Certain instruments, such as the Coulter Counter, have, as the measured size parameter, the volumes of the individual particles. Size distributions from such techniques are given in terms of spheres having the same volume as the particles of interest.

### A.3.2 Mean Free Path of Gas

The mean free path of a gas, which is the average distance that molecules travel between collisions, is an important parameter in determining the aerodynamic behavior of particles. For practical purposes, the mean free path is given with sufficient accuracy by the following equation

$$\lambda = \frac{2\mu}{1.01\text{x}10^6 P} \left( \frac{8.3\text{x}10^7 T}{3 \text{ MM}} \right)^{\frac{1}{2}} \qquad \text{(A13)}$$

where $\mu$ is the viscosity of the gas, poise,

$P$ is the pressure of the gas, atm,

$T$ is the temperature, °Kelvin, and

MM is the mean molecular weight.

### A.3.3 Slip Correction Factor

Stokes law can be applied to submicron particles if a correction factor, C, is used.

$$C = 1 + \frac{2\lambda}{D}\left[1.23 + 0.41 \exp\left(\frac{-0.44D}{\lambda}\right)\right], \tag{A14}$$

where λ is the mean free path of the gas, μm, and
D is the particle diameter, μm.

The constants in equation A14 were determined empirically for air at standard temperature and pressure, and are thus only approximate for stack conditions. If the exponential term is neglected, equation A14 is referred to as the Cunningham correction factor.

### A.3.4 Viscosity of Gas

Viscosity in fluid motion is the analog of friction in the motion of solids. It is a measure of tangential forces between layers of fluid in relative motion and the resulting dissipation of mechanical energy. In aerosol physics, the study of viscosity is concerned with the force exerted on or the energy dissipated due to a solid surface in a (relatively) moving gas stream. Gas viscosity is defined as the force per unit area a moving gas exerts on a surface divided by the net transverse velocity gradient of the gas at the surface. The unit of viscosity in the International System of Units is the pascal-second and in the cgs system is the poise [0.1 pascal-second or g/(cm-sec)]. In air and most stack gases, viscosity increases with increasing temperature.

In order to find the viscosity of the flue gas, μ, the viscosity of the pure gas components of the flue gas must first be found. Viscosity is a function of temperature, and the temperature difference in different flue gases can be quite significant. The following equations (derived from curves fitted to viscosity data from the Handbook of Chemistry and Physics, Chemical Rubber

Company Publisher, 54 Edition, 1973-1974, pp. F52-55), are used to find the viscosities of $CO_2(\mu_1)$, $CO(\mu_2)$, $N_2(\mu_3)$, $O_2(\mu_4)$, and $H_2O(\mu_5)$.

$$\mu_1 = 138.494 + 0.499T - 0.267 \times 10^{-3}T^2 + 0.972 \times 10^{-7}T^3$$
$$\mu_2 = 165.763 + 0.442T - 0.213 \times 10^{-3}T^2$$
$$\mu_3 = 167.086 + 0.417T - 0.139 \times 10^{-3}T^2$$
$$\mu_4 = 190.187 + 0.558T - 0.336 \times 10^{-3}T^2 + 0.139 \times 10^{-6}T^3$$
$$\mu_5 = 87.800 + 0.374T + 0.238 \times 10^{-4}T^2$$

where T is the temperature of the flue gas in degrees Celsius. The units of $\mu$ are $10^{-6}$ g/cm-sec. Next, these values of $\mu_1$ through $\mu_5$ are used in a general viscosity equation for a mixture of any number of components (See "A Viscosity Equation for Gas Mixtures" by C. R. Wilke, Journal of Chemical Physics, Volume 8, Number 4, April 1950, page 517) to find the viscosity of the flue gas:

$$\mu = \sum_{i=1}^{n} \frac{\mu_i}{\left[1 + \frac{1}{X_i} \sum_{\substack{j=1 \\ j \neq i}}^{j=n} X_j \phi_{ij}\right]} \tag{A15}$$

where $\phi_{ij}$ is given by the equation:

$$\phi_{ij} = \frac{\left[1 + \left(\mu_i/\mu_j\right)^{1/4} \left(M_j/M_i\right)^{1/4}\right]^2}{(4/\sqrt{2}) \left[1 + \left(M_i/M_j\right)\right]^{1/2}}, \tag{A16}$$

and $M$ = molecular weight of a component in the mixture,
$X$ = mole fraction of a component in the mixture,
$\mu$ = viscosity, g/cm-sec; $\mu_1$, $\mu_2$, etc., refer to the pure components at the temperature and pressure of mixture, $\mu$ is the viscosity of the mixture, and
$\phi$ = dimensionless constant defined above.

### A.3.5 Particle-Gas Interactions

Particle relaxation time -- For the purposes of this document, the particle relaxation time, $\tau$, may be defined as the time required for a particle to accelerate from some initial velocity to the velocity of the carrier gas.

$$\tau = \frac{2\rho_p r^2 C}{9\mu} \qquad \text{(A17)}$$

where $\rho_p$ is the particle density, gm/cm$^3$,
$r$ is the particle radius, cm,
$C$ is the slip correction factor, and
$\mu$ is the gas viscosity, poise.

Particle stopping distance -- The particle stopping distance, $\ell$, is the distance travelled by a particle as it accelerates from zero velocity to the velocity of the gas stream.

$$\ell = \tau V \qquad \text{(A18)}$$

where $\tau$ is the relaxation time (sec), and
$V$ is the velocity of the gas stream (cm/sec).

Stokes number -- The Stokes number, Stk, is the ratio of the particle stopping distance to some characteristic dimension of the sampling system. For example, if the stopping distance for particles of a given diameter is much smaller than the radius of a sampling nozzle, (Stk << 1) the particles will be sampled accurately in spite of flow disturbances due to the nozzle design or sampling velocity. If the particle stopping distance is comparable in magnitude to the nozzle diameter, however, the particles may cross flow streamlines and either enter or miss the nozzle in quantities which are not proportional to the particle concentration in the duct. Thus, for Stk on the order of 0.1 or greater, isokinetic sampling is required. In impaction theory, the characteristic dimension of the system is the radius or half width of the jet, R.

Thus $$\mathrm{Stk} = \frac{2\rho_p r^2 C V_0}{9\mu R}$$

where $V_0$ is the particle velocity, cm/sec,
$\rho_p$ is the particle density, g/cm$^3$,
$r$ is the radius of the particle, cm,
$C$ is the slip correction factor,
$\mu$ is the gas viscosity, poise, and
$R$ is the radius or half width of the jet, cm.

Particle mobility -- The ratio of the velocity of a particle to the force causing steady motion is called the mobility, b.

$$b = \frac{C}{3\pi\mu D},$$

where $\mu$ is the gas viscosity, poise,
$D$ is the particle diameter, cm, and
$C$ is the slip correction factor.

### A.3.6 Cascade Impactor Terminology

Blank -- A blank usually refers to a controlled cascade impactor test run in which the particles are removed by a prefilter. If the measured impactor stage weights are found tochange significantly and consistently, the normal runs should be corrected for this background.

Bounce -- Bounce in this document refers to inadequate retention of particles in cascade impactors which strike the impaction surface. If the particle does not adhere, it is said to bounce.

Cascade impactor -- An instrument consisting of a series of impaction stages of increasing efficiency with which particles can be segregated into relatively narrow intervals of aerodynamic diameters.

Control -- A control run is a technique which is used to confirm that variables are isolated. The control is made up to be as similar as possible to an actual run, but it is not run through the test situation. The control is then examined as would an actual test run. If the experimental variable changes significantly, the experiment is not properly set up.

Cut-point -- The cut-point of an impactor stage is the particle diameter for which all particles of equal or greater diameter are captured and all particles with smaller diameters are not captured. No real impactor actually has a sharp step function cut-point, but the theoretically defined $D_{50}$ of a stage is often called its cut-point.

$D_{50}$ -- The $D_{50}$ of an impactor stage is the particle diameter at which the device is 50 percent efficient. Fifty percent of the particles of that diameter are captured and 50% are passed to the next stage.

$$D_{50} = \sqrt{\frac{18 Stk\ \mu\ R}{C\ \rho_p\ V_j}}$$

where Stk = Stokes number, determined by calibration for 50% collection efficiency, dimensionless,

$\mu$ = gas viscosity, poise,

R = impactor jet radius (for slot impactors, the slot half width), cm,

$V_j$ = gas velocity through impactor jet, cm/sec,

C = slip correction factor, dimensionless,

$\rho_p$ = particle density, g/cm$^3$.

$D_{50}$(AI), aerodynamic impaction diameter, is found by setting C and $\rho_p = 1.0$.

$D_{50}$(A), the aerodynamic diameter, is found by setting $\rho_p = 1.0$, and

$D_{50}$(S), Stokes diameter, is found by setting $\rho_p$ = the actual particle density.

Extractive sampling -- Sampling of a particulate laden process effluent stream by means of a probe inserted inside the process stream duct to allow transport of the gas to some type of sampling instrument located outside the process stream duct.

Filter -- A mat of fibers or a porous membrane used to collect airborne particles.

Grease -- In impactor terminology, grease is a substance which is placed on an impactor stage or substrate to serve as a particle adhesive.

Impaction -- The process in which the inertia of particles in an air stream that is deflected about an obstacle causes them to strike the obstacle.

Inertial impaction parameter, $\Psi$ -- The inertial impaction parameter, $\Psi$, is similar to the Stokes number, however, the characteristic dimension of the system is the diameter or width of the jet, not the radius or half width. Thus

$$\Psi = 2CV_0\rho_p r^2/9\mu D$$

where $V_0$ is the particle velocity, cm/sec,
$\rho_p$ is the particle density, g/cm$^3$,
r is the radius of the particle, cm,
C is the slip correction factor,
$\mu$ is the gas viscosity, poise, and
D is the diameter or width of the jet, cm.

In situ sampling -- Placement of a sampling device directly into a process gas stream in order to sample the particles or gas directly.

Isokinetic sampling -- The condition in which ambient air flow has the same speed and direction as air flowing into a sampling inlet. This prevents sample bias.

Preconditioning -- Unwanted weight changes of impactor glass fiber collection substrates may be reduced by placing a large number of substrates inside the duct which is to be sampled, and pumping or passing filtered flue gas through them for several hours. Such a procedure is referred to as "preconditioning" the substrates.

Precutter or precollector -- A collection device, often a cyclone, which is put ahead of the impactor in order to reduce the first stage loading. This is necessary because in some streams the high loading of large particulate would overload the first stage before an acceptable sample had been gathered on the last stages.

Probe -- A probe is any pipe used for the transport of process effluent gas from the interior of the process ducting to a sampling instrument. Usually probes are insulated and heat traced and have some type of nozzle attachment at the end to be inserted in the gas stream for isokinetic sampling. In the case of in situ sampling, the probe is used to connect the sampling instrument inside the duct to accessory equipment outside the duct. If there is no accessory equipment, the probe is used as a handle for inserting, transversing, securing, and removing the sampling instrument.

Rebound -- Return of particles to an air stream when they fail to adhere after striking a collecting surface.

Re-entrainment -- Return of particles to an air stream some time after their deposition on a collecting surface.

Stage -- A stage of an impactor is usually considered to be the accelerating jet (or plate containing multiple jets) and the surface on which the accelerated particles impact.

Substrate -- The removable, often disposable, surface on which impacted particles are collected. Substrates are characteristically light and can be weighed on a microbalance.

Wall loss -- The deposition of particles on sampler surfaces other than those designed to collect particles. They should be collected if possible and assigned to the proper position where they would have been collected.

### A.3.7 Temperature and Pressure Standards

Laboratory standard conditions have generally been recognized for many years as 0°C and 760 mm Hg. Recently the US EPA has set standard conditions for all stationary source testing to be 20°C and 760 mm Hg. Engineering standards have been defined for some time and are 70°F and 29.92 in. Hg. In order to avoid confusion, the designation "normal", (N), is used to denote engineering standard conditions in metric units (21°C, 760 mm Hg).

When denoting measures of gas volume, the letter "d", or "D", (for "dry") is sometimes included to signify that the volume measured contains no water vapor. In stationary source testing, the letter "a", or "A", (for "actual") signifies the volume of the gas at the actual stack conditions, for example, the volume the gas would have at 200°C, 740 mm Hg, and 10% $H_2O$.

Examples of stationary source testing nomenclature:

s.d.c.f. (or DSCF or SDCF)--standard dry cubic feet--a gas volume measured at 20°C, 760 mm Hg, and 0% $H_2O$.

a.c.f. (or ACF)--actual cubic feet--a gas volume measured at conditions other than standard, usually given in the text.

ACM (or $Am^3$)--actual cubic meters

ACCM (or $Acm^3$)--actual cubic centimeters

DNCM (or DNm )--dry normal cubic meters--a gas volume measured at 21°C, 760 mm Hg, and 0% $H_2O$.

# APPENDIX B

# PARTICULATE MASS CONCENTRATION MEASUREMENTS

This appendix contains the details for conducting particulate mass concentration measurements using the EPA Reference Method 5 procedure (See Appendix E). The information is presented in four sections. Section B1 describes the equipment used in conducting these tests. Section B2 is concerned with preliminary procedures prior to sampling. Section B3 details the actual mass sampling procedures. Section B4 deals with data analysis after testing has been concluded. The sampling system described is depicted in Figure 21 of Section 2.2.2.

## B.1 GENERAL SAMPLING EQUIPMENT

### B.1.1 Temperature Measurement

Several temperature measurements are required in conducting a test for particulate mass loading, including the temperature of the stack gas, the particulate filter, and the cooled sample stream. The relative errors encountered in temperature measurements are usually small since absolute temperatures are used in all gas law calculations. In source testing, dial thermometers and thermocouples are usually used for making temperature measurements.

Two common scales are used in temperature measurement, the Celcius scale and the absolute Kelvin scale. Conversions for these are shown below for °C to °K and °F to °C.

$$°K = °C + 273°$$
$$°C = 5/9(°F - 32)$$

Mercury bulb thermometers -- The mercury bulb thermometer operates by measurement of mercury expansion with temperature increase. This expansion is linear over the range of the temperature scale. Glass mercury thermometers break easily and a risk is involved in using these in source sampling.

Dial thermometers -- Two types of dial thermometers are available for use in sampling. One is bimetallic and the other is a gas bulb thermometer. The bimetallic thermometer contains a strip of two different metals bonded together. Because of the different thermal expansion coefficients of the two metals, the bonded strip will deform with temperature, and depending on the configuration of the strip, this deformation will be transferred to a dial movement which contains a temperature scale.

Gas bulb thermometers rely on the expansion of an inert gas with temperature. This expansion is sensed as a change in pressure. The dial temperature scale is actually a pressure scale. Gas bulb thermometers are used for lower temperature ranges.

Thermocouples -- Thermocouples are the most popular device for measuring high temperatures. These consist of two dissimilar wires welded together at one junction (See Figure B1). These two wires are joined to a third wire held at a reference temperature. The difference between the temperature in question and the reference junction temperature causes an electromotive force in the system which can be sensed by a potentiometer. Several metal pair types are available. Generally Chromel/Alumel is the most common choice due to resistance to oxidation. This pair is useful from -184°C to 1260°C. Other common pairs are Copper/Constantan (-184°C to 350°C), Iron/Constantan (-158°C to 1010°C), and Platinum/Platinum 10% Rh (0°C to 1538°C).

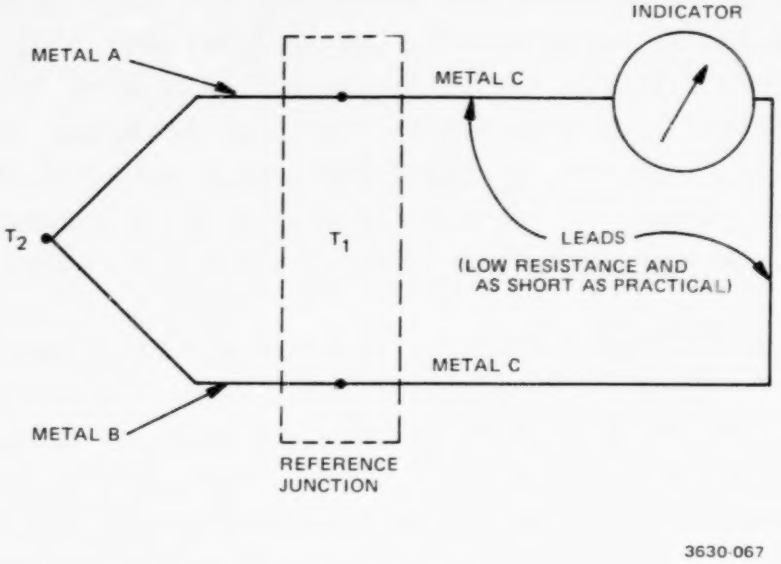

INDICATOR
METAL A
METAL C
$T_2$
$T_1$
LEADS
(LOW RESISTANCE AND
AS SHORT AS PRACTICAL)
METAL C
METAL B
REFERENCE
JUNCTION
3630-067

*Figure B1. Thermocouple junction.*

### B.1.2 Pressure Measurement

Pressure is defined as a force per unit area. Most pressure measurements are made with local atmospheric pressure as reference. The pressure above atmospheric is considered positive, and that below negative. The absolute pressure at a point is the atmsopheric plus the pressure differential.

An easy way to measure a low pressure is to balance a column of liquid against the pressure. The magnitude of the pressure can be calculated based on the measured height of the liquid column. Devices which do this are called manometers. In source sampling, manometers are often used for the determination of the stack gas velocity and the sample train flow rate. For small pressure differentials, the manometer is often inclined to increase the sensitivity. The inclined manometer is used to measure the stack velocity pressure and sample stream orifice pressure differential. It is advantageous to use manometers which have some means of protection against accidental blow out.

Mechanical pressure gauges are also available to measure low differential pressures commonly encountered, such as velocity pressure and orifice meter heads in sampling systems. The Magnehelic gauge manufactured by F. W. Dwyer Mfg. Co. in Michigan City, Indiana is an example of such an instrument. Inclined manometers, however, are generally more reliable and easier to use. They are also easier to repair.

### B.1.3 Velocity Measurement

The measurement of velocity in a duct utilizes one of the simplest devices in most sampling systems, the pitot tube. These devices are necessary because it is impossible to determine the total volumetric flow through large ducts. Only by measuring the velocity at many points and knowing the area of the duct can an accurate determination of duct volumetric flow be made. The pitot tube will not directly measure the average duct velocity but measure only the instantaneous velocity at the point at which it is located.

Several configurations are possible for pitot tubes. One, the Prandtl or standard type is shown in Figure B2. The static pressure is measured at point W. The velocity pressure is measured at point P. The velocity, $V_s$, then is given by

$$V_s = C_p \left[ \frac{2.666 \times 10^3 \left(P_p - P_w\right)}{\rho_s} \right]^{\frac{1}{2}}$$

where $V_s$ is the gas velocity, cm/sec,
$P_p$ is the measured velocity pressure, mm Hg,
$P_w$ is the measured static pressure, mm Hg,
$C_p$ is the pitot coefficient, dimensionless,
$\rho_s$ is the density of the stack gas, gm/cm³.

The pitot tube described here is usually called a standard pitot tube; the $C_p$ value for this configuration is approximately 0.99.

One configuration which does not resemble the standard pitot is the reversed or Stausscheibe (S-type) pitot tube. The S-type pitot tube is used primarily for stack testing because of one major advantage: it will not rapidly clog in gases with heavy dust concentrations. An example of one of these is shown in Figure B3. The S-type does not give the same velocity pressure as the standard pitot tube. The observed delta P is larger for a given velocity because the rear part of the tube faces downstream. This $P_s$ is a wake pressure which is lower than the static pressure. When used with a water manometer, the S-type pitot tube equation becomes

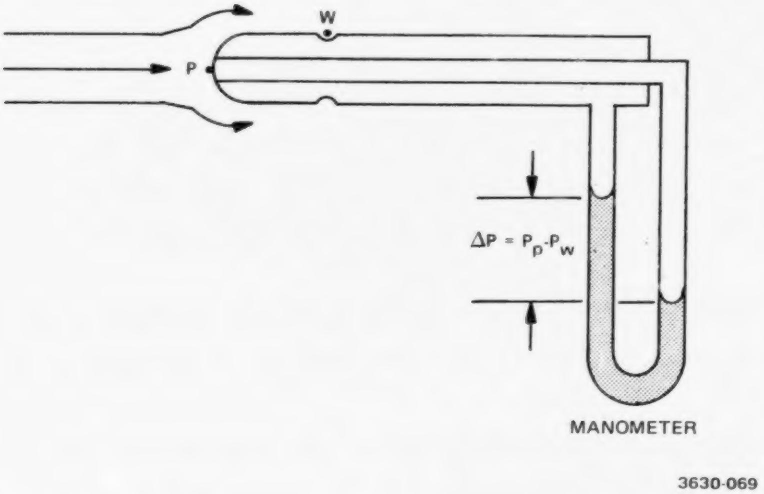
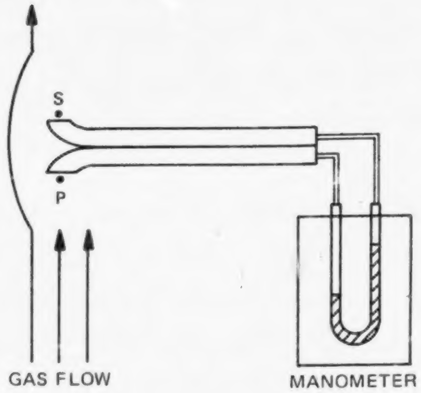

W
P
$\Delta P = P_p - P_w$
MANOMETER
3630-069

*Figure B2. Standard pitot tube.*


S
P
GAS FLOW
MANOMETER

3630-068

*Figure B3. S-type pitot tube.*

$$V_s = 101.55\ C_p \left(\frac{\Delta P}{\rho_s}\right)^{\frac{1}{2}}$$

where $V_s$ is the gas velocity, cm/sec,
$\Delta P$ is the pressure differential $(P_p - P_s)$, mm Hg,
$\rho_s$ is the density of the stack gas, gm/cm³,
$C_p$ is the pitot tube correction factor.

For the limits of 0.025 to 25.4 cm of water velocity pressure, $C_p$ for a standard (Prandtl) pitot tube usually takes on values of 0.98 to 1.00. The $C_p$ of an S-type pitot tube usually is between 0.83 and 0.87. Each must be calibrated before a test, preferably in a gas stream in which the gas properties and velocity are similar to those of the test conditions.

The calibration of a pitot tube requires a gas stream of constant and known velocity. Thus, a wind tunnel facility should be available. However, the S-type pitot tube can be calibrated against the standard pitot tube. This is the procedure designated in EPA Reference Method 2 (Federal Register, 1977).

The following equation applies in this case:

$$C_{p_{test}} = C_{p_{std}} \left(\frac{\Delta P_{std}}{\Delta P_{test}}\right)^{\frac{1}{2}}$$

where $C_{p_{test}}$ = Coefficient of the S-type pitot tube,

$C_{p_{std}}$ = Coefficient of the standard pitot tube,

$\Delta p_{test}$ = Pressure differential measured by the S-type pitot tube, mm of Hg, and

$\Delta p_{std}$ = Pressure differential measured by the standard pitot tube, mm of Hg.

To calibrate the S-type pitot tube, the velocity pressure is measured at the same point with both the S-type and standard pitot tubes. Both pitot tubes must be properly aligned in the flow field. The appropriate values are inserted in the above equation and the coefficient for the S-type pitot tube is calculated. If $C_{p_{std}}$ is not known, then a value of 0.99 should be used.

The coefficients for the S-type pitot tube should be determined first with one leg, then with the other leg pointed downstream. If the computed coefficients differ by more than 0.01, the pitot tube should not be used without proper labeling.

The determination of the average stack gas velocity, $\bar{V}_s$, is one of the greatest sources of error in stack sampling. Therefore, it is recommended that the pitot tube be recalibrated on a regular basis.

At extremely low or high velocities the pitot method is inaccurate and unreliable. There are several other mechanical and electronic methods which are available, i.e., hot wire anemometers, rotating vane anemometers and certain fluidic devices.

### B.1.4 Nozzles

The nozzle is considered the initial sampling system boundary. It removes a portion of the effluent from the duct and delivers it to the sampling probe. The nozzle has several restrictions in its use:

1. It should not disturb the duct gas stream flow.
2. It should not alter the particulate being sampled.
3. It should not add to the sample being collected.
4. It should be of a size allowing easy access.

For particulate sampling, the nozzle should disturb the gas flow as little as possible or the sample will not be representative. Any nozzle will disturb the flow, but a thin wall, sharp edged nozzle disturbs it the least. In the case of particulates, any bends in the nozzle will cause impingement of larger particles. The nozzle must then be cleaned carefully and any material found within it must be added to the total collected particulate.

### B.1.5 Probes

The probe is the sampling interface between the gas stream in the duct and the external sampling train (See Figure B4). It is exposed internally and externally to the flue gas at the nozzle end and ambient air at the exit end. The probe should not alter the sample in any way. It must be able to support itself. It must be easy to clean and it must not add to the sample. Ideally the sample should be delivered to the sample train at the stack temperature. In most instances this requires that the probe be heated to maintain the sample at stack conditions and prevent condensation. Structurally the probe must support itself, the nozzle, the pitot tube, and sometimes a thermocouple. This strength is obtained by use of a metal sheath around the probe. The requirement of probe cleaning conflicts with the structural needs. The surface of a glass tube is much more easily cleaned than a metal tube, but it is more fragile. In most cases glass probes over 2 meters in length are impractical. If a metal tube is used, some material may become trapped in the rough surface of the probe.

Glass has another advantage which should be considered. It is for all practical purposes chemically inert. This is not true of stainless steel especially if there are acid gases present in the gas stream which is being sampled.

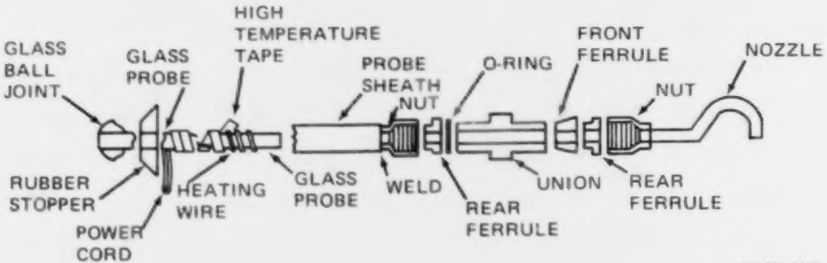

HIGH TEMPERATURE TAPE
GLASS BALL JOINT
GLASS PROBE
PROBE SHEATH
NUT
O-RING
FRONT FERRULE
NOZZLE
NUT
RUBBER STOPPER
POWER CORD
HEATING WIRE
GLASS PROBE
WELD
REAR FERRULE
UNION
REAR FERRULE
3630-070

*Figure B4. Probe and nozzle assembly.*

In the case of extremely high temperatures, the only practical choice is to use a water-cooled, high quality, stainless steel probe.

### B.1.6 Gaseous Sample Collectors

There are four main types of gas sample collection devices. One type is the cold trap which condenses vapors in the sample flow stream. Another type is that which contains a solid adsorbent. This removes the gas from the stream by surface adsorption. The third type is the grab sample container. The fourth uses the principle of gas absorption by a liquid.

Gas absorbers are generally called impingers or bubblers. Their efficiency depends on the diffusivity of the gases, the retention time in the devices, the bubble size, and the gas solubility. There are four general types of impingers; the midget impinger, the fritted glass bubbler, the modified impinger tip bubbler, and the most commonly used type, the Greenburg-Smith impinger.

### B.1.7 Particulate Sample Collectors

Filtration is the basic method for particulate collection. There are three major types of filters available today: the flat glass fiber filter, the ceramic Alundum filter, and the glass fiber bag.

Alundum thimbles are subject to variations in their particle retention efficiency as they are used because of changes in their porosity as a particulate cake forms on them. Therefore, new filters should not be used when testing relatively clean gas streams. In addition, when the thimble is used in relatively clean gas streams, only small amounts of particulate may be collected and weighing accuracy suffers. There are sometimes problems with unknown penetration characteristics of the glass fiber bags and flat filters. In many sampling trains a small cyclone pre-collector is used to remove larger particulate and allow longer sampling times.

### B.1.8 Sample Flow Rate Meters

The sharp edged orifice meter is a simple and accurate method to measure instantaneous volumetric flow rate. In source sampling it is used in conjunction with a total volume gas meter. As the gas passes through the orifice restriction, a pressure drop is created. The following equation is used for determining the flow rate through an orifice.

$$Q_m = K_m \left( \frac{T_m \Delta P_{om}}{P_m \ M_m} \right)^{\frac{1}{2}}$$

where $Q_m$ = Gas flow rate, $cm^3/sec$,
$K_m$ = Proportionality factor determined by calibration,
$T_m$ = Upstream gas temperature, °K,
$\Delta P_{om}$ = Orifice meter pressure drop, mm $H_2O$,
$P_m$ = Upstream absolute pressure, mm Hg, and
$M_m$ = Molecular weight of gas, gm/cm-mole.

For a given orifice, $K_m$ must be determined by calibration. $K_m$ is a function of Reynolds number and thus will not be a constant over the entire range of flow rates. However, for a small range of $Q_m$, such as for most sampling cases, $K_m$ is a constant. Thus, it is important to calibrate the orifice for the range of flow rates anticipated. Generally, commercial sampling trains have orifices with delta $P_{om}$ of 0-25 cm $H_2O$ over the useful flow rate range. For calibration, see Section B.1.9.

### B.1.9 Total Sample Volume

The total volume of gas sampled must be determined in most sampling trains. This provides the volume necessary to calculate the particulate concentration. Dry gas meters with capacities from 0.094-10.8 liters/sec (0.2 to 150 $ft^3$/min) are generally used. For sampling, the smallest dial face division should be 5 $cm^3$ (0.01 $ft^3$) because the meter movement is not smooth over one revolution. The dry gas meter is calibrated using a wet test meter. In a wet test meter the gas displaces water in a chamber and causes the rotor to revolve. It should be noted that the gas leaving the wet test meter is saturated with water vapor. The setup for calibrating a dry gas meter and sampling orifice is shown in Figure B5. The pertinent information should be entered on a form similar to that shown in Figure B6. The following equation is used to determine how well the dry gas meter performs as compared with the wet test meter. If the ratio defined by the equation is less than 0.99 or greater than 1.01, then the dry gas meter should be readjusted and recalibrated.

$$\gamma = \frac{V_w \, P_b \left(T_d\right)}{V_d \left(P_b + \Delta M\right) \left(T_w\right)}$$

where $\gamma$ is the ratio of accuracy of the wet test meter to the dry gas meter,

$V_w$ is the gas volume passing through the wet test meter, $cm^3$,

$P_b$ is the absolute barometric pressure, mm Hg,

$T_d$ is the average temperature of the gas in the dry gas meter, °K

$T_w$ is the temperature of the gas in the wet test meter, °K,

$\Delta M$ is the orifice meter pressure drop, mm Hg,

$V_d$ is the volume of gas passing through the dry gas meter, $cm^3$, and

$\theta$ is the time, seconds, for sampling the gas volume, $V_w$.

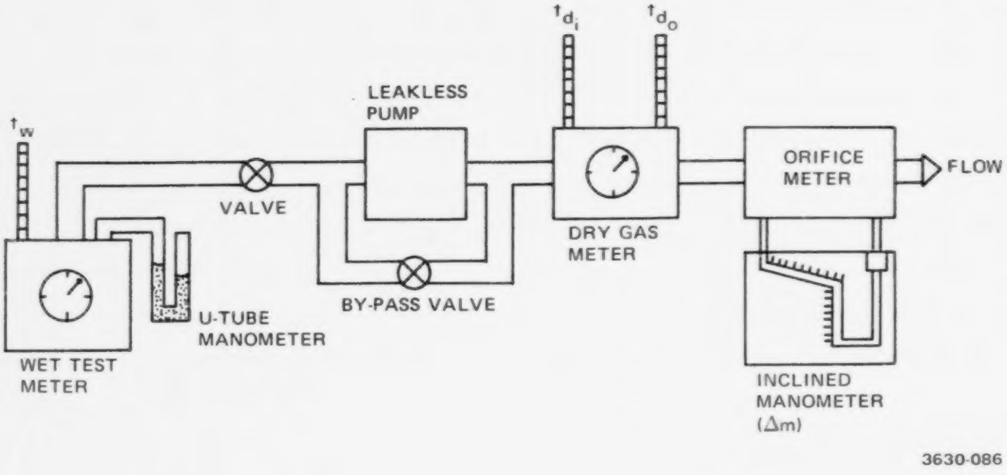

$t_{d_i}$
$t_{d_o}$
LEAKLESS PUMP
$t_w$
ORIFICE METER
FLOW
VALVE
DRY GAS METER
BY-PASS VALVE
U-TUBE MANOMETER
WET TEST METER
INCLINED MANOMETER ($\Delta m$)
3630-086

*Figure B5. Set-up for calibration of dry gas meter and orifice meter.*

DRY GAS METER NO. ____________________ ORIFICE METER NO. ____________________

DATE ____________________ WET TEST METER NO. ____________________

BAROMETRIC PRESSURE $P_b$ = __________ mm Hg CALIBRATED BY ____________________

| ORIFICE SETTING $\Delta M$ mm Hg | WET TEST METER VOLUME $V_w$ $cm^3$ | DRY TEST METER VOLUME $V_d$ $cm^3$ | TEMPERATURE | | | | TIME $\theta$ sec | $\gamma$ | $k_m$ |
|---|---|---|---|---|---|---|---|---|---|
| | | | WET TEST | DRY TEST | | | | | |
| | | | $t_w$ °K | $t_{d_1}$ °K | $t_{d_o}$ °K | $t_d$ °K | | | |
| 0.5 | | | | | | | | | |
| 1.0 | | | | | | | | | |
| 1.5 | | | | | | | | | |
| 2.0 | | | | | | | | | |
| 2.5 | | | | | | | | | |
| 3.0 | | | | | | | | | |
| 3.5 | | | | | | | | | |
| 4.0 | | | | | | | | | |
| | | | | | | | AVERAGE ➡ | | |

3630-083

*Figure B6. Data sheet for calibrating dry gas meter and orifice meter.*

The data in Figure B6 can be used to calibrate the orifice meter. In making these calculations the wet test meter is used for the flow rate, $Q_m$, and

$$Q_m = \frac{V_w}{\theta} .$$

### B.1.10 Gas Conditioning

Often the gas sample must be conditioned or treated before or while it is passed through the sampling train components. This is done either to preserve the sample or to prevent damage to the sampling train. Typical gas conditioning operations include condensing, drying, heating, and dilution.

#### Condensing

Condensers are used to remove water and other vapors from the gas sample. They work on the principle that the partial pressure of water vapor decreases with a decrease in sample temperature. For example, as indicated in stream tables, the partial pressure of water vapor at 0°C is only 0.15% of the partial pressure at 150°C. Thus, the use of an ice bath type condenser is an effective way to remove water vapor from a gas sample. This provides the moisture content which must be determined in order to calculate the molecular weight of the stack gas.

The ice bath condenser is also used in source sampling to protect other components from damage. The deposition of water vapor and water soluble constituents in such components as the dry gas meter and pump can cause severe damage.

The ice bath condenser usually consists of several wet and dry impingers connected in series, but it may be as simple as a piece of coiled tubing submerged in an ice water bath. A measured initial amount of water is put into the impinger-type condenser to assist in the condensation process. When the sampling train is operated a known amount of sample gas is passed through the system. By observing the pressure and temperature operating conditions, the amount of water condensed, and the amount of gas passing through the system, the moisture content of the gas stream can be calculated.

## Drying

In source sampling trains, gas drying is used to accomplish the same objectives as condensing. The drying operation is achieved using special chemicals which have a great affinity for water vapor. One such chemical is silica gel. The silica gel strongly adsorbs water and hence its change in weight can be used to calculate the moisture content of the gas stream. Indicating silica gel, which is granular and has a bright blue color, can be obtained commercially. As it becomes saturated with water vapor, its color changes to a light pink. If this method is to be used to determine the moisture content of a gas sample, care must be taken to insure that all particulate matter is removed first and there is no other major constituent in the gas stream which may also be adsorbed by the silica gel. The silica gel releases (desorbs) the adsorbed water vapor upon heating to 177°C and can be reused.

Often the condenser and drying tube are used in series to increase water vapor collection efficiency and obtain a high capacity for the water removed. Large mesh silica gel (6-16) is used with a filter support backing to prevent the possibility of entrainment of small particles which might damage other components.

## Heating

The heating operation is used solely to preserve the gas sample prior to passing it through the particulate collector. This is an effective method of preventing condensation of water vapor and high molecular weight substances. Therefore, it is common practice to heat sampling probes and particulate collectors such as filters to prevent deposition by condensation. If such a condensation process were allowed to occur, it would cause loss of gaseous constituents from the gas sample. In addition to causing a sampling error these materials could be deposited in inaccessible areas of the sampling system and lead to later malfunctions.

Ideally it is a good policy to try to maintain stack gas temperatures throughout the sampling train preceding the filter. However, high temperatures favor chemical reactions such as oxidation of hydrocarbons in a gas stream containing appreciable amounts of oxygen. Low temperatures, as mentioned, are conductive to condensation of water vapor and high molecular weight hydrocarbons. Hence a compromise is required and most probes and heated filter boxes operate at about 120°C. Heat sensitive sampling train components will not be affected by this temperature. Water vapor will not condense and some of the safety problems involved with the handling of hot equipment will be alleviated. Particulate compliance tests require that a gas stream temperature no higher than 120°C±14°C be maintained prior to particulate filtration. Any condensables which are taken out by the probe and filter under this condition are considered to be part of the particulate catch. A recent ruling however, allows the use of temperature up to 165°C±14°C when testing at fossil fuel utility boilers.

Dilution

Addition of a dry gas can be an effective method for preventing condensation. When this gas is added the sample is diluted. Condensation is prevented because the dry gas is capable of supporting a part of the water vapor from the gas sample even though the temperature of the mixture is reduced. The dry gas must be added in such a manner that the original sample constituents are not altered. This could be a problem with respect to particulate matter because the dilution (mixing) process could cause such events as particle agglomeration, deposition, and condensation.

### B.1.11 Pumps

The purpose of a pump is to pull the sampled gas through the sampling train components. The detail of the particular type of pump required will depend on several criteria. The pump must provide adequate flow and pressure characteristics and be durable and portable. The pump must be able to overcome the pressure drop of the other sampling train components and thereby provide the desired flow rate. It must be able to provide a wide range of flow rates as required by isokinetic sampling conditions. Often the head loss across the filter increases through the sampling tests. This puts an added burden on the pump which must still be able to maintain the required sampling rate at the nozzle tip within the stack.

The pump must be leakless when it is located ahead of the gas meter in the sampling train. If it isn't, then the metered volume will be greater than the sampled volume and hence the measured particulate concentration will be less than the true particulate concentration. The EPA Method 5 sampling train falls in this category. In many of the other sampling trains, the pump is located after the gas meters and therefore no error is involved if a leak exists in the pump.

The pump must be durable in that it is exposed to corrosive environment of the sample gas. During most source tests it is in constant operation and should be a long life component. The design should enable this component to be maintained easily; the key components should be accessible and replaceable with a minimum amount of time.

The need for portability becomes readily apparent when performing source tests, and consequently a small, lightweight pump is desirable.

There are several types of pumps suitable for source sampling trains. All are of positive displacement types which are capable of producing relatively high vacuums (∼686 mm of Hg below atmosphere pressure) and operate with a direct linear correspondence between the flow rate and inlet pressure. In commercial source sampling equipment, reciprocating diaphragm and rotary vane pumps are commonly used.

The diaphragm pump operates on the moving diaphragm principle. Gas is drawn into the chamber on a suction stroke and pushed out on the discharge stroke. On the suction stroke a suction valve is open, allowing gas to flow in. On the discharge stroke the section valve closes and a discharge valve opens allowing the gas to flow out. This intermittent operation can cause some flow fluctuation (pulsation) in the sampling train. However, this problem can be somewhat reduced by running two such pumps in parallel or by a specifically designed surge chamber in the flow line. The diaphragm in these pumps is made out of metal, rubber or plastic.

The rotary vane pump is one rotor in a casing, which is machined eccentrically in relation to the shaft. The rotor contains a series of movable vanes which seal against the pump casing. The vanes are free to slide in and out of the slots as the rotor turns. If the pump must be leakless, then only the fiber vane type pump with an oiler should be used. The oiler may have to be modified so that no ambient air leaks into the system through the oil bowl.

### B.1.12 Flow Control

Flow regulation for most sampling trains is accomplished by using a throttling valve preceding the pump. This valve varies the vacuum the pump must work against and thereby changes the flow rate. A more sophisticated arrangement uses two valves. One precedes the pump and provides a coarse control while a second one is installed in a recycle (by-pass) loop to protect the pump and provide a fine control. This latter arrangement is used in the EPA Method 5 particulate sampling train. This double valve arrangement is also easier on the pump, allowing longer pump life.

The major requirements of the flow control valve are: (a) it allows sensitive flow rate adjustment to meet proportional sampling (isokinetic) conditions and (b) it does not allow any leakage. Both these requirements depend upon the valve construction. The leakage problem poses the same potential error as was discussed for the pumps. Good quality needle valves are required in most source sampling applications.

## B.2 FABRIC FILTER SAMPLING-PRELIMINARY PROCEDURES

### B.2.1 Introduction

Before a sample is taken, several preliminary tests must be made to determine some of the characteristics of the sampling location and the gas stream. The results of these preliminary tests are the basis for determining suitability of the sampling location, the nozzle size, number of sampling points, sampling time, and nomograph settings.

### B.2.2 Physical Sampling Location Characteristics

The sampling location should be at a position where the gas flow is sufficiently uniform that an accurate sample can be obtained. Eight to ten duct diameters downstream and two duct diameters upstream from any disturbance such as bends, duct inlets, duct outlets, or changes in diameter should give sufficiently uniform flow. If the flow at the sampling location is very uniform, a minimum number of twelve sample points per sample may be used. If the upstream and downstream diameter requirement is not met, the flow at the sampling location is likely to be very non-uniform and require an increase in the number of sampling points. In such a case, Figure B7 is used as a guide to determine the number of sample points necessary to define the velocity profile adequately. Figure B7 is used by reading the number of sampling points corresponding to both the number of downstream (A) and upstream (B) diameters and selecting the greater number of sampling points. A quick pitot tube survey should indicate whether a sufficient number of points has been chosen to define the velocity profile adequately. After the number of sample points is selected, the cross section of the duct is divided into a number of equal areas as shown in Figure B8. In the case of round stacks, the sample point is located such that half of the area increment represented by that point is radially on each side of the sample point. The location of sampling points is determined as shown in Table B1. The area increments must be small enough to insure that the flow at the sampling point in each area is representative of the flow in the area; however, the total number of area increments must be limited enough so that all the points may be sampled within a reasonable period of time.

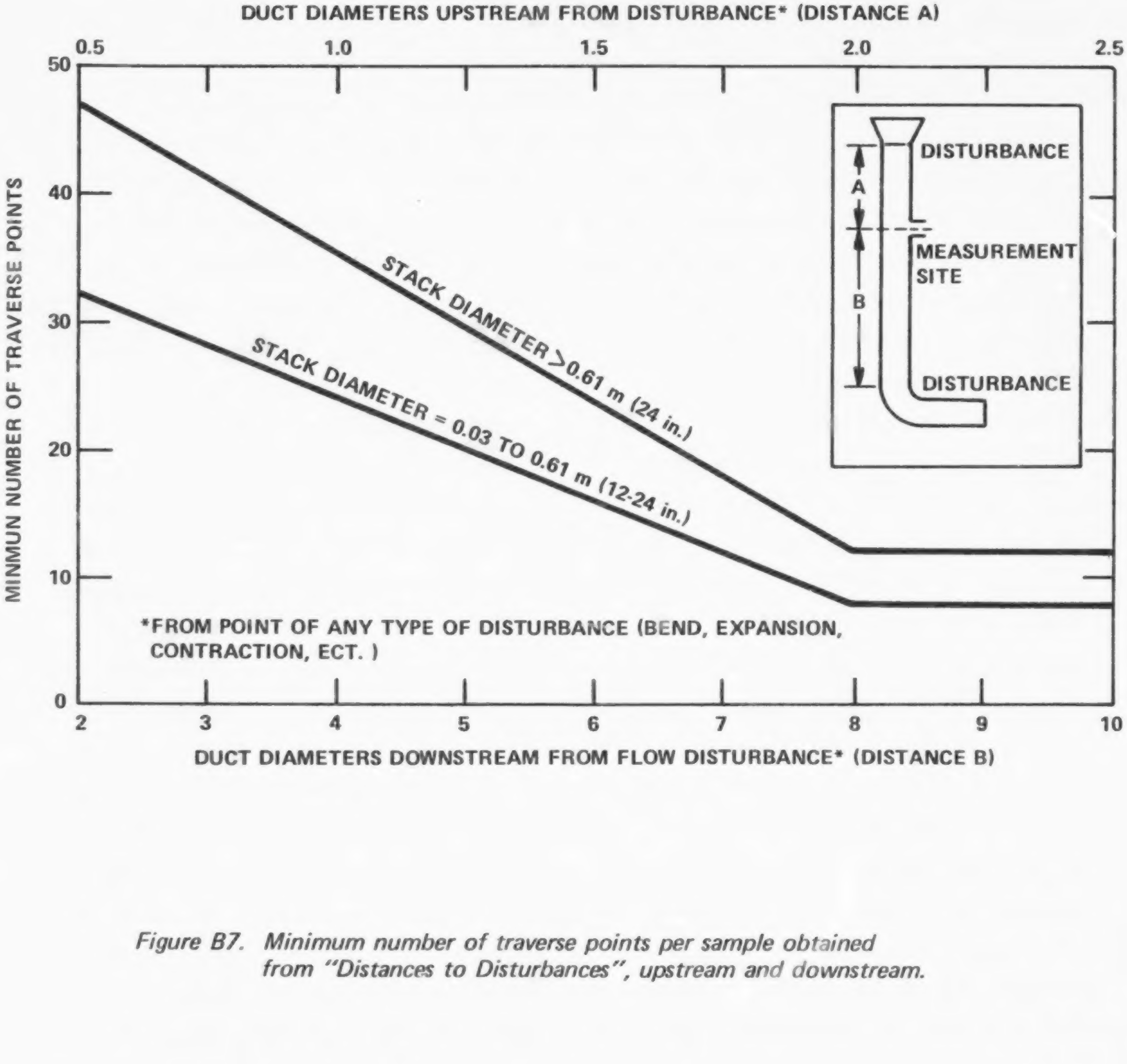


*Figure B7. Minimum number of traverse points per sample obtained from "Distances to Disturbances", upstream and downstream.*

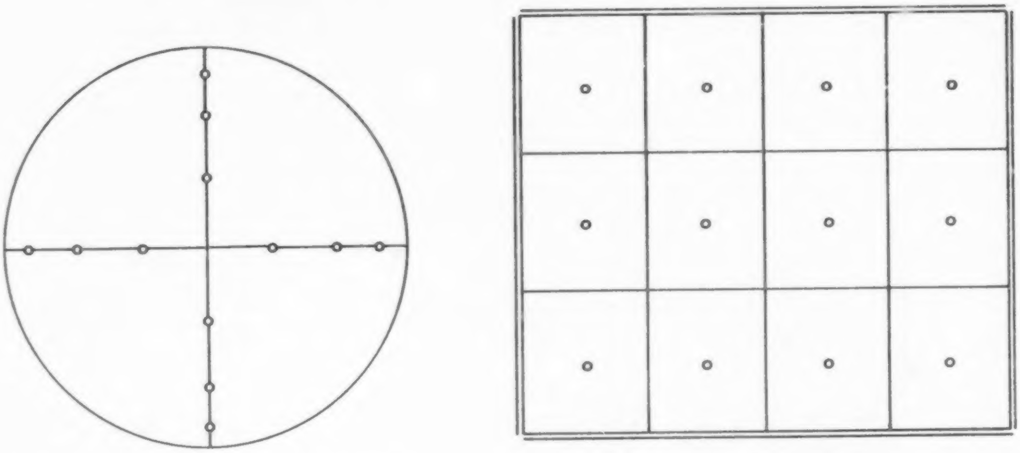
TABLE B1

Duct Traversing Length Factors

LENGTH FACTORS, $K_L$

(Fraction of stk. diam. from inside wall to traverse pt.)

| Traverse Point Number | NUMBER OF TRAVERSE POINTS ON A DIAMETER | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 2 | 4 | 6 | 8 | 10 | 12 | 14 | 16 | 18 | 20 | 22 | 24 |
| 1 | .146 | .067 | .044 | .033 | .025 | .021 | .018 | .016 | .014 | .013 | .011 | .011 |
| 2 | .854 | .250 | .147 | .105 | .082 | .067 | .057 | .049 | .044 | .039 | .035 | .032 |
| 3 | | .750 | .295 | .194 | .146 | .118 | .099 | .085 | .075 | .067 | .060 | .055 |
| 4 | | .933 | .705 | .323 | .226 | .177 | .146 | .125 | .109 | .097 | .087 | .079 |
| 5 | | | .853 | .677 | .342 | .250 | .201 | .169 | .146 | .129 | .116 | .105 |
| 6 | | | .956 | .806 | .658 | .355 | .269 | .220 | .188 | .165 | .146 | .132 |
| 7 | | | | .895 | .774 | .645 | .366 | .283 | .236 | .204 | .180 | .161 |
| 8 | | | | .967 | .854 | .750 | .634 | .375 | .296 | .250 | .218 | .194 |
| 9 | | | | | .918 | .823 | .731 | .625 | .382 | .306 | .261 | .230 |
| 10 | | | | | .975 | .882 | .799 | .717 | .618 | .388 | .315 | .272 |
| 11 | | | | | | .933 | .854 | .780 | .704 | .612 | .393 | .323 |
| 12 | | | | | | .979 | .901 | .831 | .764 | .694 | .607 | .398 |
| 13 | | | | | | | .943 | .875 | .812 | .750 | .685 | .602 |
| 14 | | | | | | | .982 | .915 | .854 | .796 | .739 | .677 |
| 15 | | | | | | | | .951 | .891 | .835 | .782 | .728 |
| 16 | | | | | | | | .984 | .925 | .871 | .820 | .770 |
| 17 | | | | | | | | | .956 | .903 | .854 | .806 |
| 18 | | | | | | | | | .986 | .933 | .884 | .839 |
| 19 | | | | | | | | | | .961 | .913 | .868 |
| 20 | | | | | | | | | | .987 | .940 | .895 |
| 21 | | | | | | | | | | | .965 | .921 |
| 22 | | | | | | | | | | | .989 | .945 |
| 23 | | | | | | | | | | | | .968 |
| 24 | | | | | | | | | | | | .989 |



CROSS SECTION OF CIRCULAR DUCT DIVIDED INTO 12 EQUAL AREAS, SHOWING LOCATION OF TRAVERSE POINTS.

CROSS SECTION OF RECTANGULAR DUCT DIVIDED INTO 12 EQUAL AREAS, WITH TRAVERSE POINTS AT CENTROID OF EACH AREA.

3630-093

*Figure B8. Examples of equal area sample points.*

In rectangular ducts or stacks, the cross section is divided into a number of equal area rectangles. The sample is taken at the centroid of each rectangular area. These areas should be laid off such that the ratio of the length to the width of the elemental areas is between one and two.

The minimum number of sample points is twelve, and the same criteria as for round ducts is used to insure that the velocity profile is adequately defined. The equivalent diameter of a square duct is approximated by 2LW/L+W where L and W are the duct cross-sectional dimensions.

In most cases the dimensions of the ducts obtained from construction drawings are accurate; however, the inside dimensions should still be measured if feasible, particularly in the case of horizontal ducts on the bottoms of which dust deposits of considerable thickness are often found. The pitot tube may be used to make this measurement but the ends should be protected to prevent material from the back wall from clogging the ends. Another critical measurement is the length of the port extension. With these measurements, the pitot tube is marked at the points to be sampled. If the outermost points are less than one inch from the walls, they should be located at one inch and noted on the sampling form. The required length of the probe (pitot tube) for each of the points may be marked with hose clamps, tape, or other suitable material compatible with flue gas conditions.

Stack pressure is determined with a leveled and zeroed manometer. The pitot tubes are aligned perpendicular to the flow stream in the stack and one of the two pitot lines is disconnected from the console. If the stack pressure is positive gauge pressure, the manometer will show positive deflection with the one pitot line connected to the positive side of the manometer. If the stack pressure is negative gauge pressure, the manometer will show positive deflection with the one pitot tube line connected to the negative side of the manometer. The stack gauge pressure ($P_S$) in millimeters of Hg is obtained by adding the stack to ambient differential pressure, $\Delta P_D$, to the ambient pressure ($P_{AMB}$).

$$P_S = \Delta P_D + P_{AMB}$$

The preliminary flue gas temperature is obtained by a suitable means such as a stem thermometer placed in the sampling port or a thermocouple with an appropriate readout device.

### B.2.3 Velocity Determination

Before velocity measurements are taken, the inclined manometer must be leveled and zeroed and must remain level during sampling. The openings of the pitot tubes should be shielded from any wind currents but not be completely closed off when the manometer is zeroed. Correct connection of the pitot tube lines may be checked by blowing gently on the upstream pitot tube opening and noting the response on the manometer. The probe is then inserted, the pitot reading noted and the pitot tube lines are switched both on the console and on the probe. If the manometer reading is the same as that prior to switching the pitot tube lines it is reasonably certain that there is no significant leak in the lines. If a leak is detected, it must be eliminated before any readings are taken.

The pitot tube lines must not be pinched or the tube stopped up during the traverse. If fluctuations in the manometer are noted, pieces of cotton or glass wool may be placed in the pitot tube lines to dampen the fluctuation but should not be packed too tightly. Since the pitot tube measures pressure differences, there is no actual air flow through the lines. If one line is completely plugged, however, the results will not be accurate. In some instances condensation of water within the pitot tube lines can cause difficulties and erroneous readings.

Once the traverse has been completed and the pressure and temperature readings have been recorded the velocity may be determined. Velocity may be calculated as follows:

$$\left(V_s\right)_{avg} = (422.67)\ C_p \left(\frac{\left(T_s\right)_{avg} (\Delta P)_{avg}}{P_s M_s}\right)^{\frac{1}{2}}$$

where $\left(V_s\right)_{avg}$ is the stack gas velocity, cm/sec,

$T_s$ is the average stack temperature, °K,

$\Delta P$ is average stack gas velocity head, mm Hg,

$M_s$ is the molecular weight of the stack gas, wet basis, gm/gm-mole,

$P_s$ is the absolute stack pressure, mm Hg, and

$C_p$ is the pitot tube correction factor.

### B.2.4 Stack Moisture Content

The stack moisture content is an important factor in stack sampling. Nozzle size selection and sampling rate are both dependent on the moisture content. A condenser method or a determination (based on the dry bulb temperature with knowledge that saturated conditions exist in the stack) are two ways of determining moisture content. A wet bulb-dry bulb technique requires less equipment but must be limited to non-acid gas streams with moisture contents of less than 15% and dew points less than 52°C. The condenser method works well for most gas streams and is relatively easy to perform.

### B.2.4.1 Condenser Method

Several condenser techniques can be used to determine stack moisture content. One such technique uses a Greenburg-Smith impinger approximately half full of water followed by a straight impinger approximately half full of silica gel. A measured volume of stack gas, usually 10 cubic feet, is drawn through the impingers at a moderate flow rate. The total change in weight of the impingers is the weight of the moisture caught. The impingers should be in an ice bath while the stack gas is drawn.

With the impinger volume increase in milliliters, $V_{wc}$, the stack gas volume in $cm^3$ ($V_m$) corrected by the dry gas meter correction factor, the absolute average meter temperature in °K ($T_m$), and the meter pressure in mm of mercury ($P_m$), the moisture fraction ($B_{wo}$) is calculated as follows:

$$B_{wo} = \frac{V_{wc}\ (1243.34\ cm^3/ml\ (H_2O))}{V_{wc}\ (1243.34\ cm^3/ml(H_2O)) + V_m \left(\frac{294.4}{T_m}\right)\left(\frac{P_m}{760\ mm}\right)}$$

### B.2.4.2 Saturation Method

If water droplets are present in the stack and the stack gas temperature is below 100°C, the gas stream may be assumed to be saturated. The moisture content is read from the saturation curve on the psychrometric chart at the stack gas temperature, Figure B9.

### B.2.4.3 Wet Bulb-Dry Bulb Method

In this method two thermometers are placed in the gas stream. One is dry and the other has a wet sock over the bulb. The temperatures are read after they stabilize. If the stack or duct pressure is near atmospheric pressure, the percent moisture may then be found from the psychrometric chart, Figure B10.

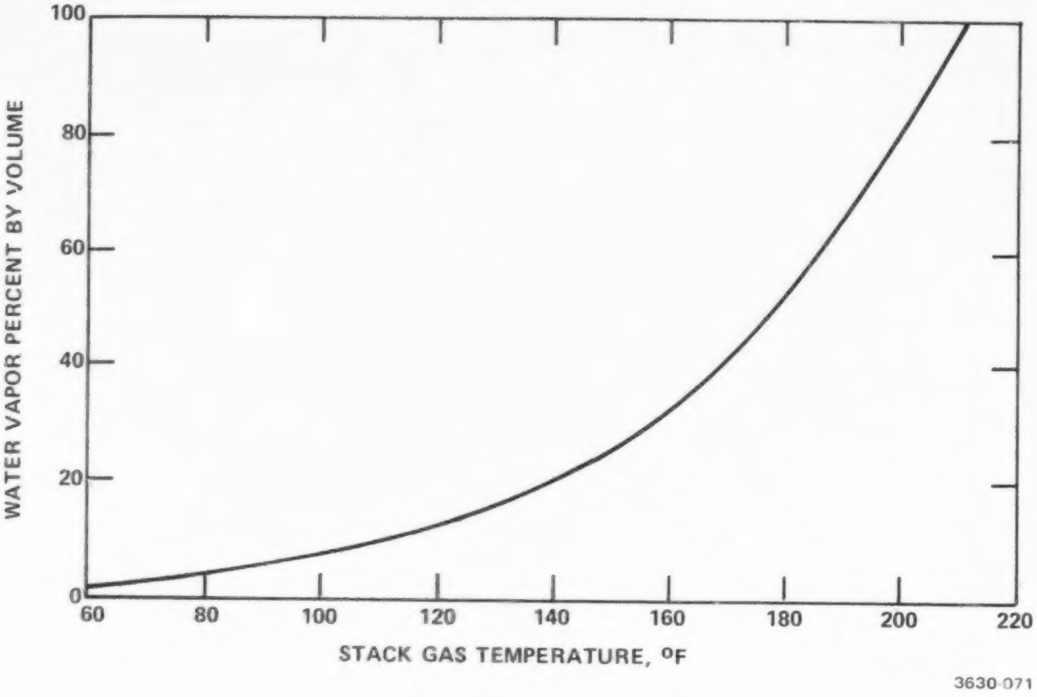


*Figure B9. Percent water vapor in air at saturation.*

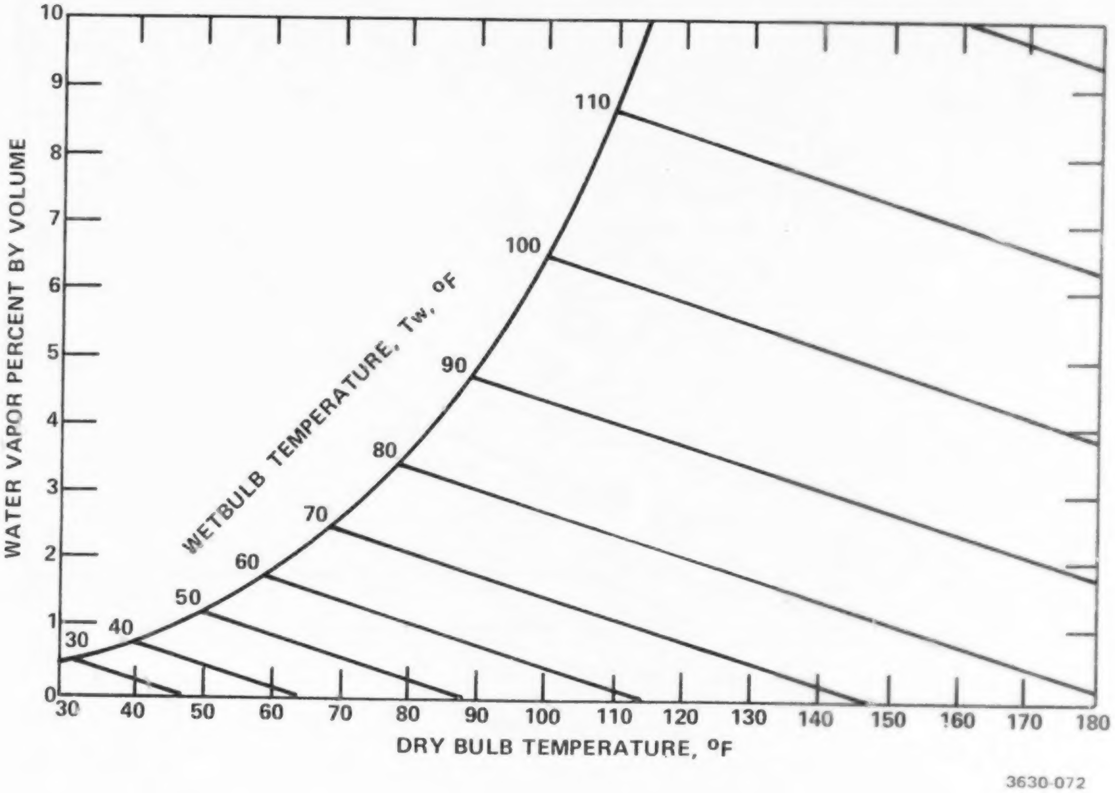


*Figure B10. Percent water vapor with wet and dry bulb.*

The percent water vapor by volume is found directly on the ordinate axis. Inputs are the dry bulb temperature on the abscissa, the wet bulb temperature, and the sloping lines which terminate at the saturated vapor line.

When obtaining wet bulb-dry bulb readings with a sling psychrometer, the plane of the thermometers should be perpendicular to the flow of gas. If it is parallel to the flow, the dry bulb should be upstream of the wet bulb. The gas velocity past the wet bulb should be from 3.7 to 9.3 meters per second. Sufficient time must be allowed for the wet bulb temperature to stabilize or inaccurate results will be obtained.

### B.2.5 Molecular Weight of the Stack Gas

The most common method of determining the composition of combustion effluents is the Orsat apparatus. Although flue gases vary in composition, they normally contain $CO_2$, CO, $O_2$, $H_2O$, and $N_2$. The Orsat analysis determines the quantities of these components (except $H_2O$) present by successive removal using suitable absorbents and measurement of the volume changes of the original sample. The Orsat analysis, as it is normally used, measures the percentage of $CO_2$, $O_2$, and CO in the sample. The difference is largely $N_2$. By changing the absorbents other components may also be measured.

After the Orsat analysis has been performed, the molecular weight of the stack gas may be determined by the following equation:

$$M_S = B_{WO}\ 18.0 + B_{DG}\ \Sigma \begin{bmatrix} \text{proportion by vol.} \\ \text{of component on} \\ \text{dry gas basis} \end{bmatrix} \times \begin{bmatrix} M_{SC} \text{ of} \\ \text{component} \end{bmatrix}$$

where $M_S$ is the molecular weight of the stack gas,

$B_{DG}$ is dry gas fraction of the stack gas,

$M_{SC}$ is the molecular weight of each component of the stack gas, and

$B_{wo}$ is the moisture fraction.

## B.3 FABRIC FILTER SAMPLING FOR PARTICULATE AND GASES

### B.3.1 Isokinetic Sampling

To obtain a representative particulate sample, the sample must be collected at a rate as nearly isokinetic as possible, i.e., the kinetic energy of the gas stream in the stack is equal to the kinetic energy of the gas stream through the sampling nozzle. Since the composition of the two gas streams is the same, this energy balance simplifies to: the velocity in the stack is equal to the velocity through the nozzle. If a particulate sample is not pulled isokinetically, inaccurate results may be obtained.

Whenever an object is placed in a moving gas stream, some disturbance of the flow patterns will occur. The purpose of isokinetic sampling is to minimize any disturbance caused by the sampling nozzle. A sample collected isokinetically through a sharp-edged nozzle, should create very little disturbance. Figure B11 illustrates this point.

Large (heavy) particles tend to travel in a straight line and are not greatly affected by flow disturbances, whereas small (light) particles tend to follow the flow lines. In a gas stream with a homogeneous distribution of large and small particles, over-isokinetic sampling will give a low particulate mass rate (PMR) because fewer large particles will be caught than are representative of the flow stream from which the gas was withdrawn. On the other hand, under-isokinetic sampling will give a high

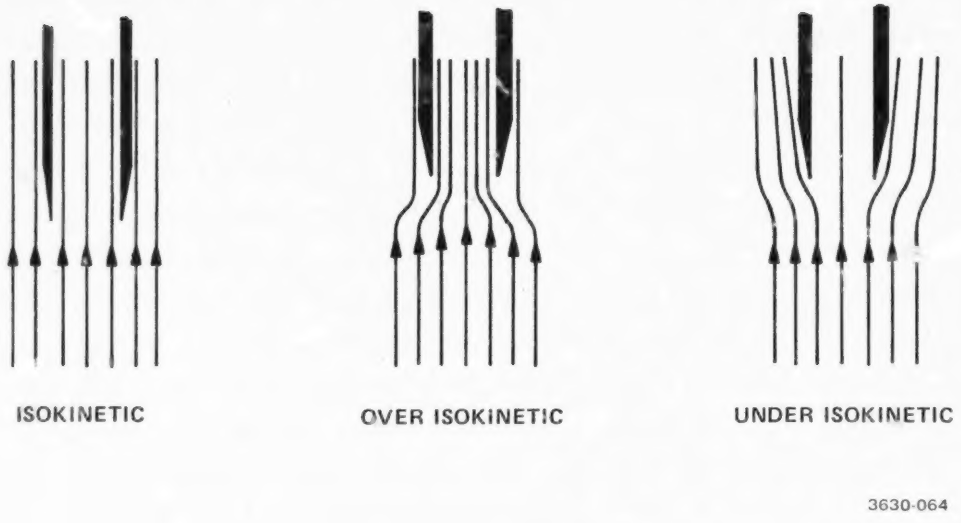

ISOKINETIC
OVER ISOKINETIC
UNDER ISOKINETIC
3630-064

*Figure B11. Isokinetic flow patterns.*

PMR due to a greater than representative number of large particles that will be caught.

The velocity of a gas stream in a stack generally varies from point to point; therefore, the flow rate of velocity through the sampling nozzle must be adjusted to maintain isokinetic conditions at each sampling point. In the sampling train, determination of the nozzle volume and the flow rate through the nozzle are based on dry gas volume and flow rate measured at approximately ambient temperatures. For this reason, the flow rate through the orifice meter which corresponds to the desired flow rate through the sampling nozzle must be determined. The stack velocity as measured by the pressure drop ($\Delta P$) across the pitot tube and the velocity through the nozzle as measured by the pressure drop ($\Delta H$) across an orifice meter at the end of the sampling train must be equal in order to maintain isokinetic flow. To speed up this calculation, two nomographs were developed by the old National Air Pollution Control Administration (NAPCA). Through the use of these nomographs, the proper size sampling nozzle may be selected and the flow adjustments required to maintain isokinetic sampling conditions may be determined. An alternate method employs the use of one of the NAPCA nomographs or a calculator for adjusting flow rates along with a mathematical method of nozzle selection. These two procedures give equivalent results.

### B.3.2 Sampling for Effluent Gases

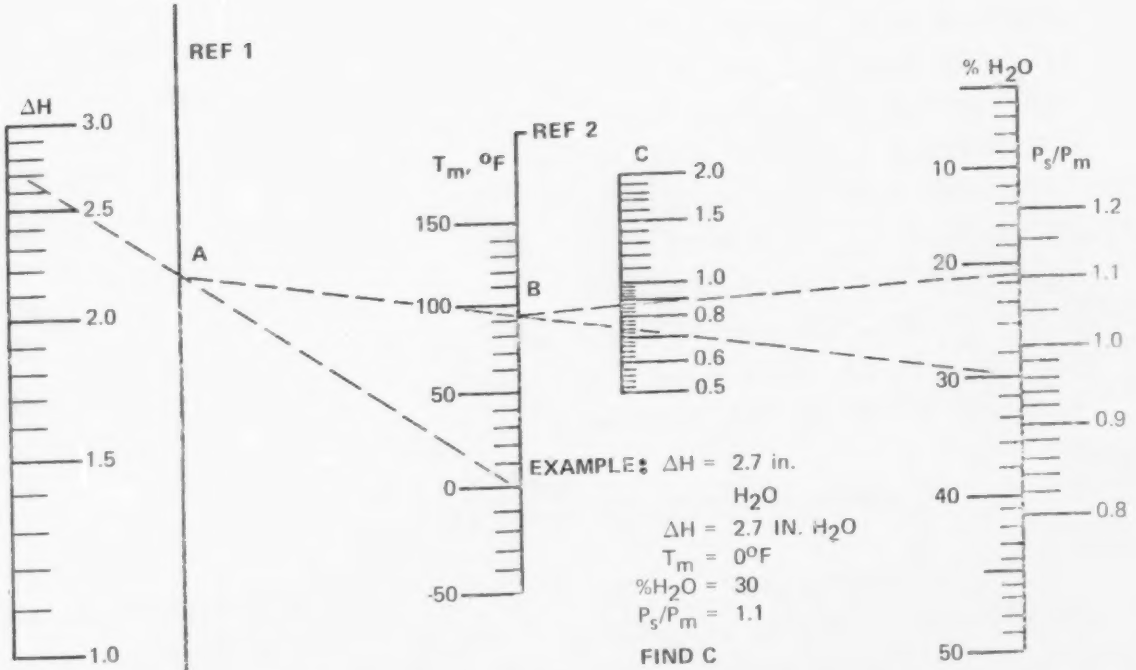
When effluent gases and particulates are to be sampled simultaneously the sampling must be performed isokinetically. It is advisable however, to choose a nozzle which will give a low volumetric flow rate to optimize gas absorption efficiency in the impinger train; 142 $cm^3$/sec (0.3 $ft^3$/min) or less is desirable. If only gases are to be sampled and if they are well mixed, isokinetic sampling is not necessary.

### B.3.3 Nozzle Selection and Nomograph Setting

Isokinetic sampling involves maintaining the flow rate through the sampling nozzle such that the velocity in the nozzle equals the velocity in the stack at the sampling point. Obviously, the flow rate through almost any size nozzle could be adjusted such that this velocity requirement is met. There are certain physical limitations, however, placed on nozzle size by the sampling equipment: pump capacity, port diameter, filter efficiency, and the critical flow through the Greenburg-Smith impingers. Another limiting factor involves the reliability of the sample. Small nozzles can yield less representative samples when large particles are present. As the ratio of nozzle tip area to stack cross-sectional area decreases, the chance of sampling at a point where the flow is not representative of the flow in the stack area that the point represents increases. The nozzle should also be at least a little larger than the largest particles that might be encountered in the stack. Some guidelines for nozzle size selection are given in the next section.

### B.3.4 EPA Reference Method 5 Procedure

Isokinetic sampling, the condition of equal velocities, implies a mathematical relationship between the two pressure drops, $\Delta P$ and $\Delta H$. The pressure drop measured by the pitot tube, $\Delta P$, indicates the stack velocity and the desired velocity through the sampling nozzle. The pressure drop across the orifice plate, $\Delta H$, represents the flow rate of dry gas through the dry gas meter. This relationship has been incorporated into two stack sampling nomographs, Figures B12 and B13. To operate the nomographs, a factor which is a composite of the test constants is obtained from one nomograph and is used to set up the second nomograph. This factor will be called "C". As the pressure drop across the pitot tube ($\Delta P$) changes from point to point, an updated desired value for the orifice pressure drop ($\Delta H$) is found from the second


REF 1
ΔH
3.0
2.5
2.0
1.5
1.0
A
REF 2
T_m, °F
150
100
50
0
-50
B
C
2.0
1.5
1.0
0.8
0.6
0.5
% H_2O
10
20
30
40
50
P_s/P_m
1.2
1.1
1.0
0.9
0.8

EXAMPLE: $\Delta H$ = 2.7 in. $H_2O$

$\Delta H$ = 2.7 IN. $H_2O$

$T_m$ = 0°F

$\%H_2O$ = 30

$P_s/P_m$ = 1.1

FIND C

DRAW LINE FROM $\Delta H$ TO $T_m$(°F) TO OBTAIN POINT A ON REF 1.
DRAW LINE FROM POINT A TO $\%H_2O$ AND READ B ON REF. 2.
DRAW LINE FROM POINT B TO $P_s/P_m$, AND OBTAIN ANSWER OF 0.85 FOR C.

3630-061

*Figure B12. Stack Sampling Nomograph (side 1).*

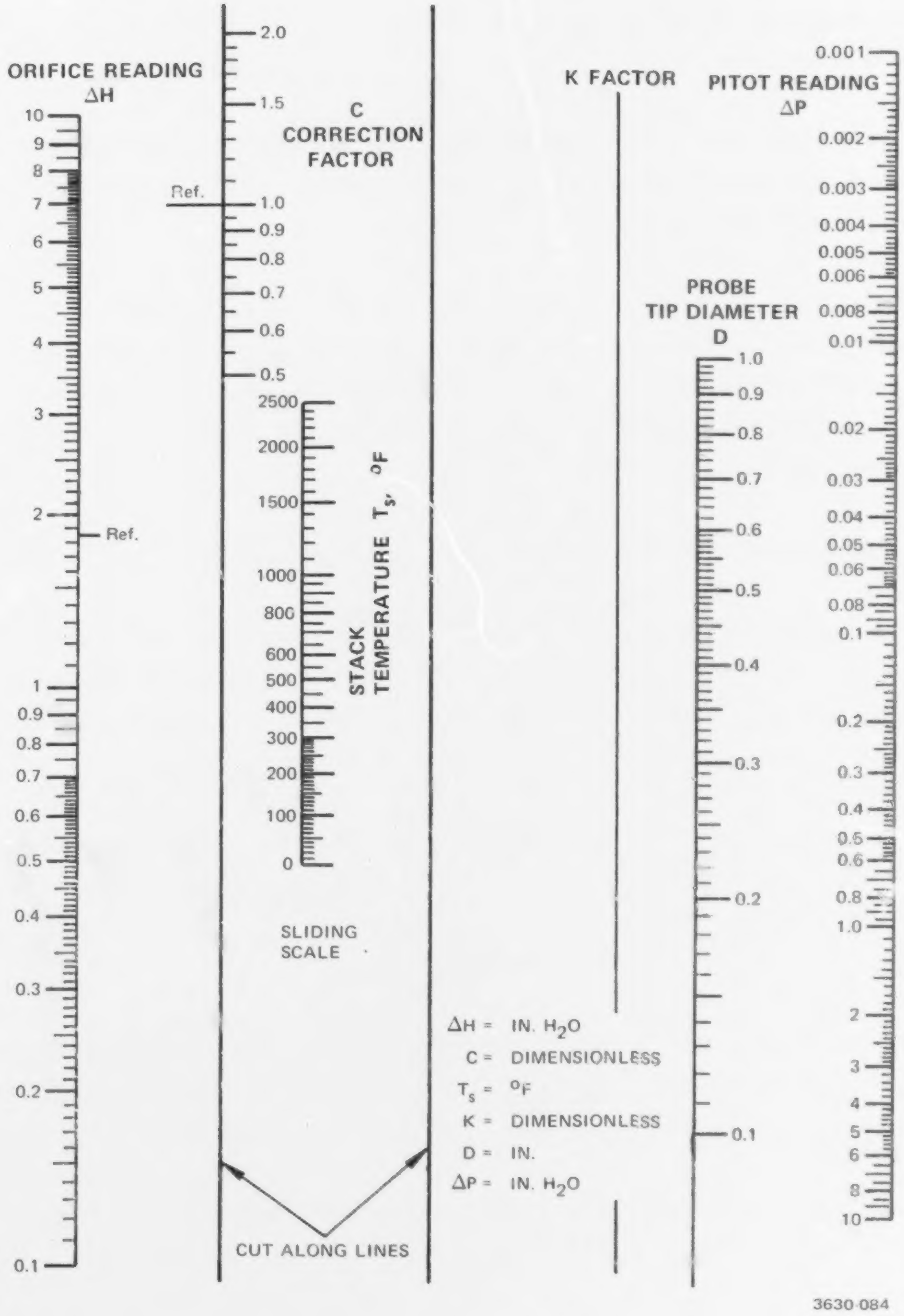

ORIFICE READING
ΔH
10
9
8
7
6
5
4
3
2
Ref.
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
Ref.
2.0
1.5
1.0
0.9
0.8
0.7
0.6
0.5
C
CORRECTION
FACTOR
2500
2000
1500
1000
800
600
500
400
300
200
100
0
STACK TEMPERATURE Ts, °F
SLIDING
SCALE
CUT ALONG LINES
K FACTOR
PROBE
TIP DIAMETER
D
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
PITOT READING
ΔP
0.001
0.002
0.003
0.004
0.005
0.006
0.008
0.01
0.02
0.03
0.04
0.05
0.06
0.08
0.1
0.2
0.3
0.4
0.5
0.6
0.8
1.0
2
3
4
5
6
8
10
ΔH = IN. H2O
C = DIMENSIONLESS
Ts = °F
K = DIMENSIONLESS
D = IN.
ΔP = IN. H2O
3630-084

*Figure B13. Stack Sampling Nomograph (side 2).*

nomograph. The flow rate may then be adjusted to give this desired pressure drop across the orifice which establishes isokinetic conditions.

In using the first nomograph, the following parameters are required to determine the "C" factor which will be carried over to the second nomograph:

1. Pressure drop ($\Delta H$) across the orifice plate when 0.75 cubic feet per minute of dry gas is flowing and the pressure and temperature are 760 mm mercury and 21.1°C respectively is determined. Note that the calibration factor of the orifice must be determined in the lab.

2. Percent $H_2O$ in the stack gas is determined prior to sampling.

3. Expected temperature at the orifice must be estimated from experience. It is usually at least ten degrees Fahrenheit higher than ambient temperature and is the same as the outlet temperature from the dry gas meter.

4. The ratio of absolute stack pressure to atmospheric pressure is also required. This ratio is approximately equal to one.

Proceed to find "C" as follows:

A. Obtain a point on the "reference one" line by connecting the orifice pressure drop, $\Delta H$, (for 0.75 cubic feet per minute) to the temperature of the orifice plate ($T_m$).

B. Draw a line from this point on the "reference one" line to the percent $H_2O$ to obtain a point on the "reference two" line.

C. Connect this point to the pressure ratio point; this line crosses the "C" scale and gives the value required to set up the second nomograph.

After the initial velocity traverse, and prior to sampling, set up the second nomograph as follows:

A. Set the vertical sliding scale so that the "C" factor mentioned above is at the reference point.

B. From the velocity traverse, calculate the average $\Delta P$. A line from this point to the stack temperature intersects the probe tip (nozzle) diameter scale.

C. From the nozzles on hand, select one near this size. Reconstruct the line through the stack temperature and use the selected probe tip diameter to give a reference point on the $\Delta P$ scale.

D. A line from the reference point on the $\Delta P$ scale to the permanent reference point on the $\Delta H$ scale locates the pivot point on the K factor line. Lock the pivot bolt that carries the clean plastic rule at this point. The nomograph is now set up for isokinetic sampling. Note that stack temperature is assumed to remain constant.

For each point of the sampling traverse the pressure drop across the pitot tube, $\Delta P$, is used as input to the nomograph. The output is the desired pressure drop across the orifice, $\Delta H$, which is required to maintain isokinetic flow. If the temperature of the stack changes appreciably during sampling, (±5% on an absolute temperature basis) the pivot point must be reset. Once the pivot point on the K factor line has been set, it is suggested that the maximum and minimum $\Delta P$'s from the velocity traverse be used to determine the range of $\Delta H$'s. This range should lie between 0.3 and 6 inches of $H_2O$. If it does not, a different choice of nozzle tip diameter should be considered.

### B.3.5 General Sampling Procedures

#### B.3.5.1 Preparing the Glassware

The glassware must be prepared and placed in the sampling box. All of the glassware should be clean and dry. The glass liner in the probe should be washed and the nozzle attached to the probe. In the sampling box, the sample first passes through a glass cyclone which has a flask attached to the bottom to catch large particles that the cyclone separates from the gas stream. The sample then goes through a fritted glass filter holder. The filter holder is 6.4 cm to 10 cm in diameter. A glass fiber filter is placed over the fritted surface. The filter should be preweighed to ±0.0001 grams. From the filter the sample goes into the condenser section. The type and number of impingers to be used in this section is dictated by the type of sample to be taken. The different impinger solutions and their uses are listed in the following paragraphs. Gummed labels can be used to label the impingers and should be placed near the top to prevent water from being absorbed from the ice slurry. The impingers should be weighed to ±0.1 gram before and after sampling. A very light coating of silicone lubricant should also be placed on the ball joints connecting the glassware to insure a vacuum-tight seal. The impinger solution volume is normally 200 ml.

#### B.3.5.2 Impinger Trains

The following tables give some recommended impinger (IMP) trains.

Preparation of Impinger Solutions

80% Isopropanol: 160 ml Technical Grade isopropanol + 40 ml deionized (DI) water.

6% $H_2O_2$: 40 ml Reagent Grade 30% Hydrogen Peroxide + 160 ml DI water.

0.1 N NaOH: 20 ml 1 N NaOH + 180 ml DI water.

1 N NaOH: 40 grams Reagent Grade NaOH (pellet form) dissolved in DI water made up to 1.0 liter volume.

Alkaline arsenite solution (0.500 N $NaAsO_2$ in 2.5 N NaOH): Dissolve 100 grams Reagent Grade NaOH (pellet form) and 32.5 grams of $NaAsO_2$ in DI water and dilute to 1.000 liter with DI water.

0.1 N $H_2SO_4$: 10 ml 2 N $H_2SO_4$ diluted with DI water to 200 ml. Prepare 2 N $H_2SO_4$ by adding 1 volume of Reagent Grade concentrated $H_2SO_4$ (18 M) to 17 volumes of DI water.

0.1 M zinc acetate: 22 grams $Zn(C_2H_3O_2)_2 \cdot 2H_2O$ in 1.0 liter DI water.

I. Particulate Only

Application: Hot mix plants, ore sintering processes, gypsum manufacturing, etc.

| IMP. NO. | CONTENTS | POLLUTANT OR COMPONENT | IMPINGER TIP CONFIGURATION |
|---|---|---|---|
| 1 | DI Water | Particulate | Straight |
| 2 | DI Water | Particulate | Greenburg-Smith (G-S) |
| 3 | Dry | Water | Straight |
| 4 | Silica Gel | Water | Straight |

II. Particulate + Sulfur Dioxide

Application: Non-ferrous smelters, Portland cement kilns, coal-fired boilers, fluid catalytic cracking units, sulfuric acid plants, boiler recovery stacks (kraft paper mills).

| IMP NO. | CONTENTS | POLLUTANT OR COMPONENT | IMPINGER TIP CONFIGURATION |
|---|---|---|---|
| 1 | 80% isopropanol | Particulate, $H_2SO_4$, $SO_3$ | Straight |

| IMP. NO. | CONTENTS | POLLUTANT OR COMPONENT | IMPINGER TIP CONFIGURATION |
|---|---|---|---|
| 2 | 6% $H_2O_2$ | $SO_2$ | G-S |
| 3 | 6% $H_2O_2$ | $SO_2$ | G-S |
| 4 | Dry | Water | Straight |
| 5 | Silica Gel | Water | Straight |

NOTES:

1. The impinger train must be purged with two cubic feet of ambient air at the end of the run to sweep $SO_2$ out of impinger #1 into impingers 2 and 3.

2. The first impinger not only is effective in trapping sulfuric acid but will also trap submicron particulate that passes through the filter.

III. Particulate + Chlorine and/or Chlorides

Application: Pulp bleaching (effluent from), Magnesium plants (drying of magnesium chloride).

| IMP NO. | CONTENTS | POLLUTANT OR COMPONENT | IMPINGER TIP CONFIGURATION |
|---|---|---|---|
| 1 | DI Water | HCl, $Cl^-$<br>Particulate | G-S |
| 2* | Alkaline Arsenite Solution | HCl, $Cl_2$<br>Particulate | G-S |
| 3 | Alkaline Arsenite Solution | HCl, $Cl_2$ | G-S |
| 4 | Dry | Water | Straight |
| 5 | Silica Gel | Water | Straight |

*NOTES: It is important to measure the volume of this solution as accurately as possible since the analysis will be based on the molar quantity of arsenite remaining.

IV. Particulate in the Presence of Hydrogen Fluoride

Application: Alumina reduction plants and phosphate fertilizer manufacturing (acidulation process).

| IMP NO. | CONTENTS | POLLUTANT OR COMPONENT | IMPINGER TIP CONFIGURATION |
|---|---|---|---|
| 1 | Water | Particulates, inorganic fluoride particulate | G-S |
| 2 | Water | Particulates, inorganic fluoride particulate | G-S |
| 3 | Dry | Water | Straight |
| 4 | Silica Gel | Water | Straight |

NOTE: Use filter bypass instead of fritted filter.

V. Particulate + Ammonia

Application: Effluent from manufacturing, prilling and drying of ammonium nitrate or ammonium phosphate fertilizer.

| IMP NO. | CONTENTS | POLLUTANT OR COMPONENT | IMPINGER TIP CONFIGURATION |
|---|---|---|---|
| 1 | Water | Particulate | G-S |
| 2 | 0.1 N $H_2SO_4$ | $NH_3$ | Straight |
| 3 | 0.1 N $H_2SO_4$ | $NH_3$ | Straight |
| 4 | Dry | Water | Straight |
| 5 | Silica Gel | Water | Straight |

NOTE: Water is used in the first impinger in order that particulate may be measured. The contents of this impinger will be saturated with ammonia. Partial recovery of ammonia from the first impinger may be accomplished by purging the impinger train with approximately 2 $ft^3$ of ambient air.

VI. Particulate + Hydrogen Sulfide

A. Application: Carbon black plants (furnace effluent), lime kilns (kraft paper mills).

| IMP NO. | CONTENTS | POLLUTANT OR COMPONENT | IMPINGER TIP CONFIGURATION |
|---|---|---|---|
| 1 | 0.1 M zinc acetate | $H_2S$ | G-S |
| 2 | 0.1 M zinc acetate | $H_2S$ | G-S |
| 3 | Dry | Water | Straight |
| 4 | Silica Gel | Water | Straight |

B. Application: kraft pulp mills, boiler recovery stacks.

| IMP NO. | CONTENTS | POLLUTANT OR COMPONENT | IMPINGER TIP CONFIGURATION |
|---|---|---|---|
| 1 | Water | Particulate<br>$SO_3$, $H_2SO_4$ | Straight |
| 2 | 0.1 M zinc acetate | $H_2S$ | G-S |
| 3 | 0.1 M zinc acetate | $H_2S$ | G-S |
| 4 | Dry | Water | Straight |
| 5 | Silica Gel | Water | Straight |

NOTE: With a conventional sampling train $H_2S$ and $SO_2$ cannot be determined simultaneously.

### B.3.5.3 Checking the Sampling Train for Leaks (Vacuum Check)

A vacuum-tight system is necessary to prevent any dilution air from being pulled into the sampling line. After the probe and sample box are connected and suspended from the monorail or other support, the umbilical cord containing the sample line, pitot tube lines and thermocouple leads may be connected. The inlet side of the cyclone is then sealed for the vacuum check. The pump is then started with the coarse valve closed and bypass valve open. As the coarse valve is slowly opened the vacuum will begin to increase. The bypass valve is slowly closed until the vacuum reaches 381 mm gauge. At this vacuum the flow rate through the dry gas meter should not exceed 9 $cm^3$/sec (0.02 $ft^3$/min). If a leak is present all connections should be checked to eliminate the leak and the above procedure repeated. The seal on the cyclone must be removed slowly before the pump is turned off to prevent liquid backup in the impingers. After the above vacuum check is performed the probe liner is connected to the cyclone and a vacuum check on the total system may be performed. The pump is started again and the coarse valve is slowly opened. After the flow starts, the nozzle tip is sealed and vacuum should start to build in the system. When the sample line vacuum reaches

381 mm the coarse valve should be closed. The vacuum should hold steady if there are no leaks. If it holds for about fifteen seconds, the nozzle tip is opened and the vacuum should drop. Then the pump is turned off with care not to back up liquid in the impingers. Under no circumstances should the pump be turned off while the nozzle tip is sealed. The power line is then plugged in and the heater turned on. The probe heater, if used, should be connected and the probe heater turned on. The pitot tube manometer may then be zeroed by shielding the end of the pitot tube from any wind or disturbance. The ends should not be plugged, however. The orifice manometer should also be zeroed. The manometer must be level at this time and throughout the sampling period. The pitot tube should also be checked to be sure the lines are hooked up properly. The man on the platform can blow lightly on the upstream pitot tube while the console operator checks the manometer displacement. Before sampling begins, the console operator should be sure the manometers are leveled and zeroed, the temperature indicator is working properly, and the nomograph or calculator is properly set up.

The console operator should have decided on how long to sample at each point. The number of sample points is determined by generally accepted rules, e.g., velocity traverse, but the sampling time is based on knowledge of the plant operations and the approximate particulate loading which may be obtained from plant personnel on a pretest. The sample collected on the filter should be large enough to weigh accurately and be representative of the conditions in the stack; however, care must be exercised to prevent the filter from clogging, or in wet stacks, the impingers from filling up. It is preferable to sample for at least an hour. If the process is cyclic and portions of the cycle give high particulate mass rates (PMR) which are not upset conditions, these portions of the cycle must be included in the sample. The sample size should be at least 850 liters at standard conditions. If conditions permit, some adjustments in nozzle size and sample flow rate can often be made to satisfy time requirements. It is usually best to sample each point for no less than three minutes as this allows time for adjusting flows and recording data.

### B.3.5.4 Sampling

When the console operator is ready and the probe and sample box have heated sufficiently, the initial dry gas meter reading is recorded and the sampling probe is pushed carefully into the duct to the point nearest the back wall. This allows the probe to cool in hot stacks as it comes out, shortening the time required for cooling after the sample is taken. This also allows the use of the stack heat to help heat the probe. The nozzle must not hit the back wall or the inside of the port where deposited material might contaminate the sample. If this procedure is followed, the last point sampled will be the point nearest the port. As soon as the probe is positioned the operator should record the time and start the run by turning on the pump, opening the coarse valve and adjusting the bypass valve until the desired flow rate calculated from the nomograph or K factor is obtained. The data should then be recorded. The probe crew should be notified 15 to 30 seconds before the probe is to be moved and the signal to move to the next point is given approximately 5 seconds before that time. When the probe is repositioned, the operator should read the new $\Delta P$, use the nomograph or calculator, adjust the flow rate, and record the data required on sampling field data sheets. The opening at the port should be plugged to prevent dilution or abnormal distortion of the flow patterns in the stack. After the last point has been sampled, the operator turns off the pump and records the meter reading. If more than one port is to be sampled, the sampling box is transferred to the next port and the above procedure repeated. The probe is then removed carefully from the port so the open end of the nozzle does not hit the port. The probe should also be kept horizontal and the nozzle plugged as soon as possible after it is removed from the stack to prevent loss of sample. In some cases condensate will collect in the probe and if the probe is tipped, some of the sample might be lost. The particulate trapped in the probe can represent a significant portion of the total sample. Normally three sample runs are taken. Each run normally consists of two traverses. Each run is considered as a separate sample, and the calculations are performed for each sample. The results of the runs are then averaged.

Before making a second run the percent isokinetic of the first run should be checked. The nomograph or calculated K factor should be changed and an additional sample taken if the percent isokinetic varies by more than ±10% from 100%. (See Data Reduction Section.)

### B.3.5.5 Sample Handling

At the end of each sample run the electrical power is disconnected and the hot side of the sample box is opened to allow the glassware to start cooling. The pitot tube lines to the probe are disconnected and the probe is removed from the box and the ends plugged as soon as possible. The impingers may now be removed and weighed. After the impingers are weighed, the liquid in the impingers is placed in clean sample bottles to be taken to the lab. Some particulate or condensable compounds will occasionally get past the filter and be collected in the liquid. The sample catch from the filter and the probe washings should be collected and stored separately from the impinger catch. The probe and nozzle are washed carefully and the washings collected in a clean sample bottle to be taken to the lab, evaporated and then weighed. The cyclone and flask and the connecting glassware are washed and the washings added to the probe washings. The weight of any water caught in the cyclone must be determined and added to the impinger weight gain. The filter is removed from the holder, folded with the particulate side in and placed in an envelope to be taken to the lab and weighed. The filter holder is washed and the washings added to the probe wash. Usually distilled water is used to wash the glassware, but in some instances, acetone may be used. Precautions must be taken to eliminate the possibility of tampering with, accidental destruction of, and/or physical and chemical action on the samples.

To reduce the possibility of invalidating the results, all components of the sample should be carefully removed from the sampling train and placed in sealed, nonreactive, numbered containers. The samples are then delivered to the laboratory for analysis. It is recommended that this be done on the same day that the samples are taken. If this is impractical, all samples should be placed in a carrying case (preferably locked) in which they are protected from breakage, contamination, and loss.

Each container should have a unique identification to insure positive identification and to preclude the possibility of interchange. The identification of the container should be recorded on the analysis data sheet so it will be associated with the sample throughout the test and analysis.

The samples should be handled only by persons associated in some way with the task. A good general rule to follow is "the fewer hands the better", even though a properly sealed sample may pass through a number of hands without affecting its integrity.

## B.4 MASS CONCENTRATION DATA REDUCTION

After performing particulate mass measurements on the inlet and outlet of a fabric filter, the data must be reduced to obtain particulate emissions concentrations and other pertinent parameters.

To illustrate these calculations, an example of data collected on a single test will be presented along with the appropriate calculations to obtain the necessary results.

From the particulate source test data given in Table B2 we find:

Orifice $\Delta P_{avg} = 3.69$ mm Hg

$T_{m_{avg}} = 23.6°C = 296.9°K$

B.4.1 Volume of Gas Sampled

$$V_{mc} = V_{m_{std}} = V_m \frac{T_{std}\left(P_{bar} + \Delta H_{ave}\right)}{T_m \, P_{std}}$$

$$= 2.8 \; \frac{294}{297} \; \frac{(749 + 3.69)}{760}$$

$$= 2.75 \text{ DNM}^3$$

where $V_{mc}$ is the volume of gas sampled through the dry gas meter at standard conditions (21°C, 760 mm Hg), $\text{DNM}^3$.

$V_m$ is the volume of gas samples through the dry gas meter at meter conditions, $m^3$.

$T_{std}$ is 21°C, 294°K.

$T_m$ is the average dry gas meter temperature, 23°C, 297°K.

$P_{bar}$ is the uncorrected barometric pressure at the outlet of the orifice meter, 749 mm Hg.

$\Delta H_{ave}$ is the average pressure drop across the orifice meter, 3.69 mm Hg.

$P_{std}$ is the absolute pressure at standard conditions, 760 mm Hg.

## TABLE B2

## EXAMPLE - PARTICULATE SOURCE TEST DATA - BAGHOUSE INLET

General

Firm - CM & S Public Service
Source - Coal Boiler #1
**Sampling Location - Baghouse Inlet**

Personnel - K.M.C., J.D.M., & W.B.S.

Date - 3/18/77
Test No.-5
Witnessed by - D.B.H.

Equipment

Gas Meter No.-3
Sample Box No.-8

Probe No.-3 Length-305 cm Cp-.87

Nozzle Dia. - 0.89 cm

Filter No. 212 Silica Gel No. 87
Probe Wash Sample No. - KC10

Conditions

Orifice ΔH - 3.74 mm Hg
Assumed %$H_2O$ - 8% $T_s$ - 149°C

Assumed $\Delta P_{AVG}$-1.5 mm Hg $P_s/P_m$-1.0

Assumed $T_M$-26.7°C $A$ - 9.29 m²

Bar Pressure - 749 mm Hg
C-Factor - .860
Probe Heater Set - 135°C
Filter Oven Set - 121°C

| Traverse Pt. | Time (sec) | Dry Gas Meter ($\times 10^{-3} m^3$) | Pitot ΔP mm Hg | Orifice ΔH mm Hg | Meter Inlet °C | Temp. Outlet °C | Stack Temp. °C | Pump Vacuum mm Hg |
|---|---|---|---|---|---|---|---|---|
| 1 | 300 | 53.5 | .99 | 4.52 | 23 | 20 | 146 | 152.4 |
| 2 | 300 | 244.4 | 1.08 | 4.89 | 24 | 20 | 146 | 152.4 |
| 3 | 300 | 415.7 | 1.18 | 4.86 | 24 | 21 | 150 | 152.4 |
| 4 | 300 | 677.4 | 0.45 | 4.30 | 24 | 22 | 150 | 177.8 |
| 5 | 300 | 929.5 | 1.53 | 3.31 | 25 | 22 | 145 | 177.8 |
| 6 | 300 | 1088.3 | 1.62 | 3.16 | 25 | 22 | 147 | 152.4 |
| 7 | 300 | 1311.5 | 1.77 | 2.97 | 25 | 22 | 151 | 152.4 |
| 8 | 300 | 1613.4 | 1.76 | 2.80 | 25 | 22 | 151 | 152.4 |
| 9 | 300 | 1928.6 | 1.55 | 3.29 | 26 | 22 | 153 | 127.0 |
| 10 | 300 | 2137.9 | 1.18 | 3.10 | 27 | 23 | 150 | 152.4 |
| 11 | 300 | 2331.3 | 1.12 | 3.38 | 28 | 23 | 147 | 177.8 |
| 12 | 300 | 2560.1 | 1.08 | 3.74 | 28 | 24 | 145 | 177.8 |
| Totals | 3600 | 2800 | | | | | | |
| Averages | | | 1.28 | 3.69 | 25.3 | 21.9 | 148.4 | |

Stack Pressure -7.5 mm Hg

Test End - 12:00 noon
Test Start-11:00 a.m.

Impinger Outlet Max Temperature 10°C

| $CO_2$ | $O_2$ |
|---|---|
| 11.0% | 6.0% |
| 11.5% | 5.5% |
| 12.0% | 5.8% |

Condensate Collected
#1 100 ml
#2 50 ml
#3 10 ml

Silica Gel Mass Charge - +46 grams

$\Delta H_{AVE}$=3.69 mmHg

Filter and Probe Wash Catch - 150 mg

### B.4.2 Volume of $H_2O$ Vapor in Stack Gas

Water condensed in impingers = 160 ml, water absorbed on silica gel = 46 grams.

$$V_{wc} = V_{l_c} \frac{\rho H_2O}{M_{H_2O}} \frac{R\ T_{std}}{P_{std}} = 206\ \frac{1.00}{18.0}\ \frac{(6.2383\text{x}10^4)\ (294)}{760} = 0.28\ m^3$$

where $V_{wc}$ is the total volume of water in the sampled gas at standard conditions (21°C, 760 mm Hg), $m^3$.

$V_{l_c}$ is the total volume of liquid $H_2O$ collected in impingers and on silica gel, 206 ml.

$\rho_{H_2O}$ is the density of water at standard conditions, 1.00 g/ml.

R is the ideal gas constant, $6.2383 \times 10^4$ mm Hg $cm^3$/gm mole-°K.

$T_{std}$ is the standard temperature, 21°C, 294°K.

$P_{std}$ is the standard pressure, 760 mm Hg.

$M_{H_2O}$ is the molecular weight of water, gm/gm-mole.

### B.4.3 Moisture Content of Stack Gas

$$B_{wo} = \frac{V_{wc}}{V_{m_{std}} + V_{wc}} = \frac{0.28}{2.75 + 0.28} = 0.0924 \text{ or } 9.24\%$$

where $B_{wo}$ is the mole fraction or proportion by volume of $H_2O$ vapor in the stack gas.

$V_{wc}$ is 0.28 $m^3$ $H_2O$ vapor at standard conditions.

$V_{m_{std}}$ is 2.75 $m^3$ of dry sampled gas at standard conditions.

### B.4.4 Molecular Weight of Stack Gas

From Orsat analysis per EPA Method 3:

$CO_2$ ave = 11.5%
$O_2$ ave = 5.8% by volume (Dry basis)
CO ave = 0%

From moisture analysis per EPA Method 5:

$H_2O$ avg = 9.24%

From EPA Method 3:

$$\% N_2 \text{ (dry basis)} = 100\% - (\% CO_2 + O_2 + \% CO) = 100\% - (17.3\%) = 82.7\%$$

$$M_w = \left(1-B_{wo}\right)\left(B_{CO_2}M_{CO_2} + B_{O_2}M_{O_2} + B_{CO}M_{CO} + B_{N_2}M_{N_2}\right) + B_{wo}M_{H_2O}$$
$$= (1-.0924)\ [(0.115)(44.0) + (0.058)(32.0) + (0.0)(28.0) + (0.827)(28.0)] + (0.0924)(18.0) = 28.96 \text{ gm/gm-mole}$$

where $M_w$ is the molecular weight of the stack gas, gm/gm-mole, wet basis.
$B_i$ is the mole fraction of the component gas.
$M_i$ is the molecular weight of the component gas.
$B_{wo}$ is the mole fraction of the water vapor in the stack gas.

### B.4.5 Excess Combustion Air (per EPA Method 3)

$$\%EA = \frac{(\%O_2) - 0.5(\%CO)}{0.264(\%N_2) - (\%O_2) + 0.5(\%CO)} \times 100$$

$$= \frac{(5.8) - 0.5(0)}{0.264(82.7) - (5.8) - 0.5(0)} = 36\%$$

where %EA is the percent excess combustion air.

$\%O_2$ is the percent $O_2$ by volume on a dry basis.

$\%N_2$ is the percent $N_2$ by volume on a dry basis.

%CO is the percent CO by volume on a dry basis.

0.264 is the ratio of $O_2$ to $N_2$ in air by volume.

### B.4.6 Particulate Emissions Concentration

$$C'_s = \frac{M_n}{V_{m_{std}}} = \frac{150.0}{2.75} = 54.55 \text{ mg/DNM}^3$$

where $C'_s$ is the concentration of particulate matter in the stack gas, mg/DNM$^3$.

$M_n$ is the total particulate mass collected on filter media, 150.0 mg.

$V_{m_{std}}$ is the volume of stack gas sampled (volume through dry gas meter), DNM$^3$.

### B.4.7 Stack Gas Volumetric Flow Rate (per EPA Method 2)

$$\left(V_s\right)_{avg} = K_p C_p \left(\sqrt{\Delta p}\right)_{avg} \left[\frac{T_{s\ avg}}{P_s M_w}\right]^{\frac{1}{2}}$$

where $\left(V_s\right)_{avg}$ is the average stack gas velocity, m/sec.

$K_p$ is $128.83 \frac{m}{sec} \left(\frac{gm}{gm\text{-}mole\text{-}\ K}\right)^{\frac{1}{2}}$

$C_p$ is the pitot tube coefficient.

$(T_s)_{avg}$ is the average stack gas temperature, °K.

$\left(\sqrt{\Delta p}\right)_{avg}$ is the average of the square roots of velocity heads of stack gas, in $H_2O$, determined according to EPA Methods 1 and 2.

$P_s$ is the absolute stack gas pressure, mm Hg.
$M_w$ is the molecular weight of the stack gas, gm/gm-mole (wet basis).

From Table B2:

$$\left(\sqrt{\Delta p}\right) \text{avg} = 1.13$$

$$C_p = 0.860$$

$$\left(T_s\right) \text{avg} = 421.7°K$$

$$P_s = P_{bar} + \left(P_s\right) = 749 + (-0.75)$$

$$= 748.25 \text{ mm Hg}$$

$$M_w = 28.96 \text{ gm/gm-mole}$$

$$\left(V_s\right)_{avg} = (128.83)(0.860)(1.13) \left[\frac{421.7}{(748.25)(28.96)}\right]^{\frac{1}{2}}$$

$$\left(V_s\right)_{avg} = 17.47 \text{ m/sec}$$

Then calculating the average stack gas volumetric flow rate:

$$\bar{Q}_s = 3600 \frac{\text{sec}}{\text{hr}} \left(1-B_{wo}\right)\left(V_s\right)_{avg} A_s \frac{T_{std} P_s}{\left(T_s\right)_{avg} P_{std}}$$

where $\bar{Q}_s$ is the average volumetric flow rate at dry standard conditions, DNM$^3$/h

$B_{wo}$ is the mole fraction of water vapor in the stack gas, dimensionless, and

$A_s$ is the cross-sectional area of the stack at the sample point, 9.29$^3$m (from the Particulate Test Data Sheet).

Then,

$$\bar{Q}_s = (3.600 \times 10^3)(.9076)(17.47)(9.29)\,\frac{(294)(748.25)}{(421.7)(760)}$$

$$= 3.64 \times 10^5 \text{ DNM}^3/\text{h}$$

Therefore, the particulate inlet mass emission rate can be calculated by $\overline{PMR}_s = \bar{Q}_s\, C_s$

where $\overline{PMR}_s$ is the average particulate inlet mass emission rate, (mg/hr calculated by the concentration method).

$$\overline{PMR}_s = 3.64 \times 10^5\,\frac{\text{DNM}^3}{\text{hr}} \cdot 54.55 \text{ mg/DNM}^3$$

$$= 1.986 \times 10^7 \text{ mg/hr of particulate.}$$

B.4.8 Average Isokinetic Ratio

$$I_{avg} = \frac{(V_n)_{avg}}{(V_s)_{avg}} \times 100$$

where $I_{avg}$ is the average percent isokinetic.

$(V_n)_{avg}$ is the average gas velocity into the nozzle entrance.

$(V_s)_{avg}$ is the average gas velocity of the stack gases.

Writing the equations for the nozzle velocity independent of the velocity pressure measured in the stack.

$$(V_n)_{avg} = \frac{V_{mc}\,\dfrac{T_s P_m}{T_m P_s M_d}}{A_n\,\theta}$$

where $V_{n\ avg}$ is the average nozzle velocity.
$V_m$ is the volume of gas through the dry gas meter, $DNM^3$ (dry).
$T_s$ is the average absolute stack gas temperature °K.
$P_s$ is the average absolute stack pressure, mm Hg.
$P_m$ is the average absolute dry gas meter pressure, mm Hg

$$(P_m = \Delta m + P_{bar})$$

$T_m$ is the average absolute meter temperature, °K.
$M_d$ is the average mole fraction of the dry stack gas, $(1-B_{wo})$.
$A_n$ is the area of the nozzle, $m^2$.
$\theta$ is the total sampling time, sec.

Then,

$$V_{n\ avg} = \frac{(2.8)\ \frac{421.7}{294}\ \frac{752.69}{748.21}\ \frac{1}{.9076}}{(6.22 \times 10^{5} m^2)\ (3600\ \text{sec})} = 19.88\ \text{m/sec}$$

$$I_{avg} = \frac{V_n\ avg}{V_s\ avg} = \frac{19.88}{17.47} = 1.14 \text{ or } 114\%$$

To conform with the federal performance standards, the average isokinetic rate $I_{avg}$, must be $>0.90$ and $<1.10$. In this example, since $I_{avg}$ does not fall within these limits, this test would be discarded.

### B.4.9 Mass Collection Efficiency Calculation

This example has shown calculations for a test at a baghouse inlet. Similar calculations are made on data taken at baghouse outlets. After determining the inlet and outlet mass loading concentrations, the fabric filter efficiency can be calculated from

$$\%EFF = \frac{PMR_{Inlet} - PMR_{Outlet}}{PMR_{Inlet}} \times 100\%$$

$PMR_{Inlet}$ = Particulate inlet mass emission rate.
$PMR_{Outlet}$ = Particulate outlet mass emission rate.

# APPENDIX C
# CASCADE IMPACTOR SAMPLING TECHNIQUES

This appendix contains detailed guidelines for the operation of cascade impactors to measure the particle size distributions at the inlet and outlet of fabric filters. These instructions are taken from "PROCEDURES FOR CASCADE IMPACTOR CALIBRATION AND OPERATION IN PROCESS STREAMS", EPA-600/2-77-004, by D. B. Harris. Minor modifications have been made to make them specifically applicable to fabric filter evaluations, and a more detailed description of data reduction techniques and data presentation formats has been added.

## C.1 THE PRESURVEY

The key to performing a successful fractional efficiency evaluation is thorough planning based on a complete pretest site survey. The survey should provide adequate information at as low a cost as possible. Some sites will require more information and some less. As far as is possible, the information noted during the presurvey should be measured rather than obtained from plant records or personnel.

As the presurvey is generally conducted by one or two men "traveling lightly," the apparatus used during the presurvey should be as light and compact as possible. A presurvey sample train is shown in Figure C1. This system can be built into a single, suitcase-size package, and serves well as a presurvey sample train. The impactor which is to be used during the main test program should normally be used during the presurvey. This

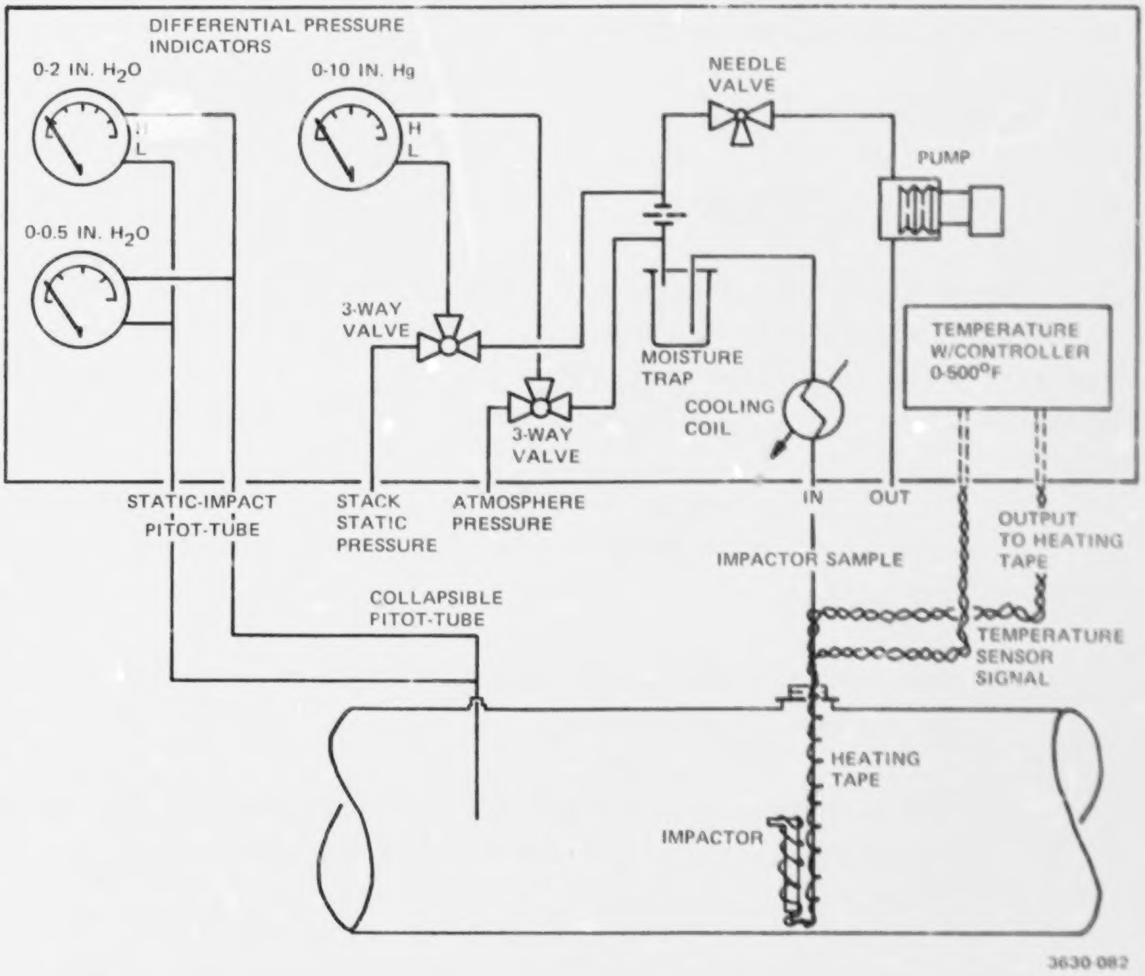

DIFFERENTIAL PRESSURE INDICATORS
0-2 IN. $H_2O$
H
L
0-10 IN. Hg
H
L
NEEDLE VALVE
PUMP
0-0.5 IN. $H_2O$
3-WAY VALVE
MOISTURE TRAP
TEMPERATURE W/CONTROLLER 0-500°F
COOLING COIL
3-WAY VALVE
STATIC-IMPACT PITOT-TUBE
STACK STATIC PRESSURE
ATMOSPHERE PRESSURE
IN
OUT
OUTPUT TO HEATING TAPE
IMPACTOR SAMPLE
COLLAPSIBLE PITOT-TUBE
TEMPERATURE SENSOR SIGNAL
HEATING TAPE
IMPACTOR
3630-082

*Figure C1. Presurvey sampling with a cascade impactor.*

is because the suitability of substrates and adhesives must be checked out. These problems are discussed more fully in later sections.

In general, the presurvey work should be done using the techniques described in this manual. Less precision is required, but the accuracy must be high enough to provide useful information in designing the test program. The decisions which must be made are summarized in Table C1.

## C.2 EQUIPMENT SELECTION

### C.2.1 Impactor Selection

The selection of the proper impactor for a particular test situation is primarily dependent upon the mass loading of the gas stream and its effect on sampling time. There are three major criteria to be met to match an impactor to a particulate stream.

1. The sampling period must be long enough to provide a reasonable averaging of any short term transient in the gas stream.

2. The loading on a given impactor stage must be low enough to prevent re-entrainment.

3. The sampling rate through the impactor must be low enough to prevent scouring of impacted particles by high gas velocities.

For these reasons, an impactor with a comparatively low sample rate must be used in a gas stream with a high mass loading. The low sample rate allows a longer sampling time, although in some situations it will still be undesirably short. Conversely,

## TABLE C1. IMPACTOR DECISION MAKING

| Item | Information Required | Criteria |
|---|---|---|
| Impactor | Loading and size estimate | a. If particle concentration below 5.0 µm is less than 0.46 gm/am$^3$ (0.2 grain/acf), use high flow rate impactor. |
| | | b. If particle concentration below 5.0 µm is greater than 0.46 gm/am$^3$ (0.2 grain/acf), use low flow rate impactor. |
| Sampling rate | Loading and gas velocity | a. Fixed, near isokinetic |
| | | b. Limit so last jet velocity does not exceed:<br>60 m/sec greased<br>35 m/sec without grease |
| Nozzle | Gas velocity | a. Near isokinetic, ±10% |
| | | b. Sharp edged; min 1.4 mm ID |
| Pre-cutter | Size and loading | If pre-cutter loading is comparable to first stage loading--use pre-cutter. |
| Sampling time | Loading and flow rate | a. Per Figure C4 |
| | | b. No stage loading greater than 10 mg |
| Collection substrates | Temperature and gas composition | a. Use metallic foil or fiber substrates whenever possible |
| | | b. Use adhesive coatings whenever possible |
| Number of sample points | Velocity distribution and duct configuration | a. At least two points per station |
| | | b. At least two samples per point |

in a low mass loading situation such as a control device outlet, a high sample rate device must be used if a significant amount of sample is to be gathered in a reasonable amount of time.

A cascade impactor can normally yield useful information over a range of sample rates differing by a factor of 2 or 3. As high efficiency control devices cause the outlet mass loading to differ from the inlet by a factor of $10^3$, the same impactor can seldom be used on both inlet and outlet. Both high and low flow rate impactors are usually required to determine the fractional efficiency of fabric filter installations. Some commercial impactors are constructed such that their stage and nozzle configurations can be altered, and they can serve as either high or low sample rate impactors. Others are fixed with respect to sample rate.

### C.2.2 Sample Trains

Figure C2 is a flow diagram of a typical impactor sampling train. As shown, it is desirable to have the impactor inside the stack with a straight nozzle. The various parts of the sample train are discussed below.

A sampling probe leading to an impactor outside of the duct should be used only if absolutely necessary. The probe should be as short as possible and contain the fewest possible bends. It is recommended that a pre-cutter cyclone be mounted at the probe inlet to remove particles larger than approximately ten micrometers and thus reduce line losses.

Heating system -- The criteria for heating are given in Section C.4.3. If heating is required, the entire impactor must be either wrapped in a heating tape or put in a custom-fitted heating mantle. The temperature control should be based on the

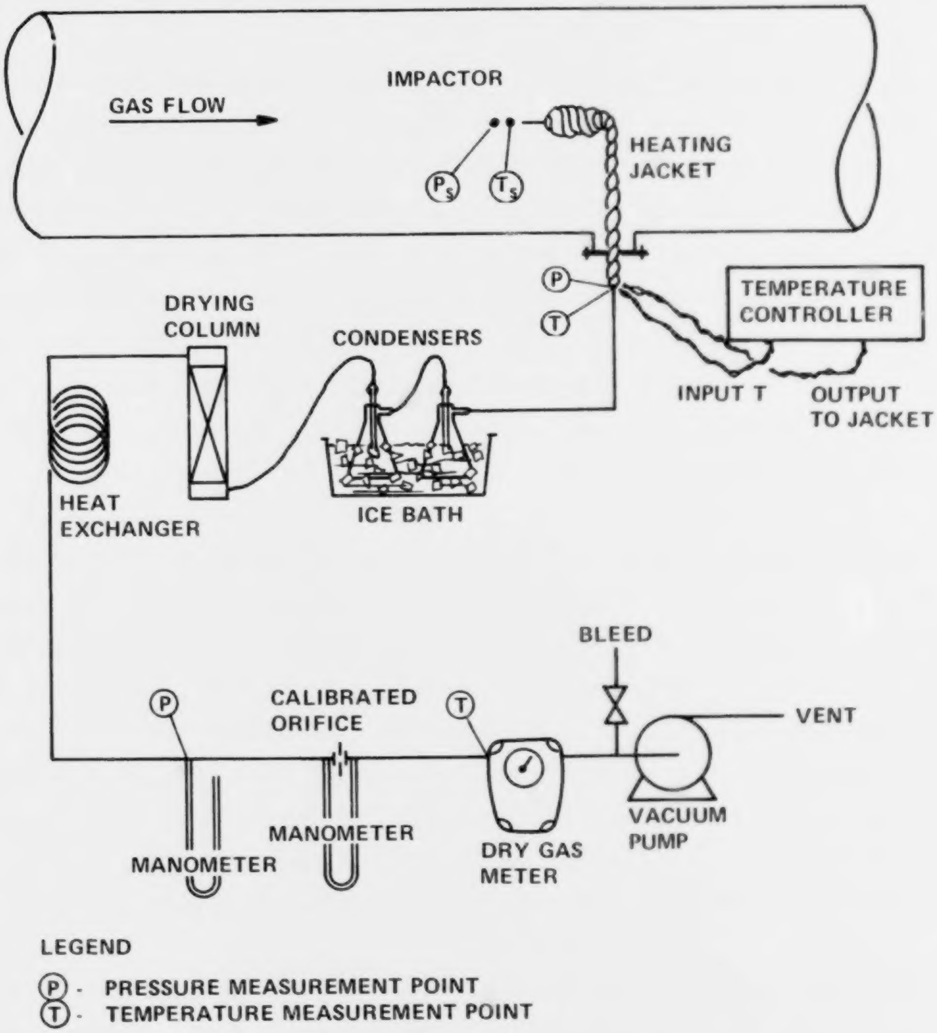

IMPACTOR
GAS FLOW
HEATING JACKET
$P_s$
$T_s$
P
T
TEMPERATURE CONTROLLER
DRYING COLUMN
CONDENSERS
INPUT T
OUTPUT TO JACKET
HEAT EXCHANGER
ICE BATH
BLEED
P
CALIBRATED ORIFICE
T
VENT
MANOMETER
MANOMETER
DRY GAS METER
VACUUM PUMP
LEGEND
P - PRESSURE MEASUREMENT POINT
T - TEMPERATURE MEASUREMENT POINT
3630-081

*Figure C2. Typical sample train with a heated impactor.*

temperature at the outlet end of the impactor. Often the temperature is measured between the last stage and back-up filter. The impactor temperature can be controlled either manually or automatically. An automatic controller has usually been found to be worth the money by releasing the operator for other tasks.

Flue gas conditioning -- It is usually necessary to cool and dry the flue gas before it reaches the flow measuring section. Condensation in the orifice would distort the measurement; also, it is useful to protect the equipment from the condensate, which, in $SO_2$-containing gases, is likely to be sulfuric acid. The type of condensers shown are usually satisfactory. Packed bed drying columns are commercially available. The heat exchange coil is used to bring the gas temperature to essentially ambient so that there will not be significant temperature gradient across the flow measuring device.

Flow measurements -- At least two flow measuring devices are used in series. Normally, a calibrated orifice is used in conjunction with a dry gas meter, as shown. At very low sample rates the dry gas meter may be inaccurate. The commonly used diaphragm-type positive displacement gas meter becomes increasingly inaccurate at flow rates less than five percent of rated capacity. For a typical stack sampling gas meter this would be approximately 23.3 $cm^3/sec$ (0.05 cfm). Another calibrated orifice or a rotameter should then be used as the second flow meter.

Vacuum pumps -- The vacuum pump should usually be placed at the end of the sample train. This is because vacuum pumps tend to leak and all of the flow measurements must be made upstream of any leak. The flow rate can be controlled by using an inlet side air bleed or with a recirculating bypass.

Pressure measurements -- Most of the pressure measurements are made with manometers, but calibrated differential pressure meters are equally acceptable. The in-stack pressure needed is the static pressure, which is not exactly the downstream pressure of an S-type pitot tube. A true static pressure measurement should be made. It is not necessary that this be part of the impactor train, but it can be.

The pressure at the downstream end of the impactor, between the last stage and the final filter, must be known. It can be measured, but this is often inconvenient. If a flow rate pressure drop calibration is available for the impactor (without final filter), it is normally acceptable to calculate the pressure drop. Correction must be made for pressure and temperature differences between the calibration conditions and the actual conditions.

The pressure at the inlet to the metering devices must be known. In the system shown in Figure C2, the pressure is metered ahead of the calibrated orifice and the orifice pressure drop is used to calculate the pressure going into the dry gas meter. The dry gas meter pressure should be measured if there is a reason to think the procedure above was not adequate.

Temperature measurements -- It is necessary to know the temperature at all points where the pressure is measured. Any convenient device of known accuracy can be used to make the measurements. The measurement in-stack can easily be made at the probe end with a thermocouple. The temperature at the downstream end of the impactor is made directly behind the final filter and is used to control the heating tape if one is used. If the heat exchanger in the train brings the gas temperature to about ambient, only one temperature reading will be necessary at the flow meters. This is usually most conveniently done at the dry gas meter, as taps are available on the meter.

### C.2.3 Balance Requirements

For the accurate weighing of the collected material a balance with a sensitivity of at least 0.05 mg is required. This is especially true for the lower stages of the low sample rate impactors where collection of 0.3 mg or less is not uncommon. The balance must also be insensitive to vibration if it is to be used in the field. It is also desirable to have a balance with a large weighing chamber. These capabilities are available in several electrobalances marketed in the United States.

More information on selected sampling train components can be found in Appendix B, Section B.1.

## C.3 IMPACTOR SUBSTRATES

### C.3.1 Collection Substrates

For reasons which have been discussed, very accurate determinations of impactor stage catch weights are necessary. Impactor stages are generally too heavy for the tare capacity of field-usable precision balances. Thus, a substrate which can be weighed on the balance is used. Generally, these substrates are made of metal foil or glass fiber.

Glass fiber substrates -- Glass fiber substrates are used on some commercial impactors. In addition to providing a lightweight impaction surface, glass fiber mats greatly reduce re-entrainment due to particle bounce. They are superior to greased metal substrates in very high temperature applications where the greases tend to evaporate. Care must be taken when using glass fiber substrates in streams containing sulfur dioxide, however. Recent experimentation (Felix, et al, 1977) has shown that glass fiber materials often exhibit anomalous weight gains due to sulfate uptake on the substrates. Apparently, sulfur dioxide in a gas stream can react with basic sites on most glass fiber materials and form sulfates.

There are two approaches to alleviate this problem. Substrates which will gain weight from sulfate uptake can be preconditoned in the flue gas before weighing. Two to six hours of exposure to the flue gas will suffice where mass loadings are high and sample times are short. In the situations where sample times are long and the collected amount of particulate matter is small, it may be necessary to condition the substrates for as long as twenty-four hours to eliminate significant sulfate uptake and weight gains. Repeated weighings to check weight gains are necessary to confirm that the substrates can be used. Another approach is to use a fibrous substrate which shows little weight gain in a sulfur dioxide stream, if one can be found. It should be noted that the particle retention characteristics of different fiber materials vary, and the impactor calibration could change significantly if the substrate is changed. In the report on substrate experiments (Felix, et al, 1977), a laboratory preconditioning procedure is described in detail.

Greased substrates -- "Grease" must often be used on metal foil substrates to improve their particle retention characteristics. This is particularly important with hard, bouncy particles. Impactor stage velocities of 60-65 m/sec have been used on greased substrates with good results, while particle bounce can become a problem at about half of that rate on ungreased substrates.

Finding a suitable grease can be difficult. The grease should not flow at operating temperature, and it must be essentially nonvolatile. Gas chromatographic materials such as polyethylene glycol 600 have exhibited more consistent characteristics than materials such as stopcock grease. Another class of materials which may be suitable are high vacuum greases; Apiezon L and H in particular have performed well at temperatures up to 120°C. The greased substrates must be tested as blanks in filtered process gas before they are used in the test program.

The greases are normally applied as suspensions or solutions of 10-20 percent grease in toluene or benzene. The mixture is placed on the substrate with a brush or eyedropper, baked at 204°C for 1 to 2 hours, and then desiccated for 12 to 24 hours prior to weighing. It is important to avoid an excess of grease. The desiccated, greased substrate should be tacky, but not slippery, with a film thickness about equal to the diameter of the particles which are to be captured.

Horizontal operation of the impactors with greased substrates is not recommended due to possible flow of the grease. Care must also be taken to ensure that grease is not blown off the substrates (which tends to occur at jet velocities greater than 60 m/sec). To some degree, grease blow-off can be avoided by using a light coating of grease on the last stages. This is normally satisfactory from an adhesive standpoint, as the last stages usually have the lightest loading along with the highest jet velocity. Inspection of the stage catches is the best way to check on this problem.

### C.3.2 Back Up Filters

Back up filters are used on all impactors to collect the material that passes the last impaction stage. Binderless glass fiber filter material is normally used for this purpose in all the impactors, although the exact configuration varies.

Glass fiber back up filters have the same problems as do glass fiber substrates. Their use in process gases containing sulfur dioxide is suspect, and blanks must be run to check out the problems. Pure Teflon filters may alleviate this problem if they can be used.

## C.4 PREPARATION AND SAMPLING

### C.4.1 Substrate Preparation

It is assumed that the substrates have been properly prepared and that the necessary quality assurance steps have been taken. The substrates should be carefully weighed and kept in a desiccator until they are to be placed in the impactor.

### C.4.2 Impactor Orientation

Whenever possible, the impactor should be oriented vertically to minimize gravitational effects such as flow of grease or fall-off of collected particles. Sampling situations requiring horizontal placement will occur, and extra care must be taken on such occasions not to bump the impactor against the port during entry or removal.

### C.4.3 Heating the Impactor

All condensable vapors must be in a gaseous state until they exit from the impactor, unless a condensate is the prime aerosol being measured. In gas streams above 177°C, auxiliary heating should not be required. Below 177°C the exit temperature of the impactor should be maintained at least 10°C above the process temperature if condensable vapors are present. A thermocouple feedback temperature controller has proven useful.

When condensable vapors are present, it is sometimes necessary to heat the impactor probe to prevent any condensate formed in the probe from entering the impactor and contaminating the substrates. Water vapor is the primary problem. The probe temperature should be maintained above the vapor's dewpoint.

Whether the impactor is being heated in the duct or externally with heater tape, an allowance of 45 minutes warmup time is recommended as a minimum to ensure that the impactor has been heated to duct or operating temperature. Thermocouple monitoring of the impactor temperature and gas temperature is recommended.

### C.4.4 Probes

Sampling probes leading to an impactor outside of the duct should be used only if there is no other way. They should be as short as possible and contain the fewest possible bends. It is recommended that a precutter be mounted at the duct end of the probe to remove the large (>10 μm) particles and thus reduce line losses.

### C.4.5 Nozzle and Sampling Rate Selection

It is preferable to use as large a nozzle diameter as possible to minimize sampling errors resulting from nozzle inlet geometry. When very small nozzles have been used with the Brink impactor, there have been some cases in which large amounts of material were retained in the nozzle or the nozzle was completely blocked. It is recommended that the inlet nozzle not be smaller than 1.4 mm, and some types of particulate material may require a larger minimum nozzle size. In some instances bent nozzles are necessary due to port location and gas direction, but these should be avoided. Problems occur in cleaning bent nozzles, and it is difficult to determine the size interval in which the deposited material originated. If they cannot be avoided, bends should be as smooth as possible and of minimum angle in order to minimize the losses in the fine particle region.

For hard, "bouncy", particulate, the sampling rate must be such that the last stage velocity does not exceed 60 m/sec for greased collection surfaces or 35 m/sec for ungreased plates if no suitable substrate can be found to limit particle bounce. The flow rates above should not be considered the final word on nozzle velocity. Particle bounce has been observed at nozzle velocities as low as 10 m/sec. Some particulate materials are "sticky" and will adhere at well above the maximum velocity for hard particles. The exposed substrate should be visually examined for evidence of re-entrainment and the rates adjusted accordingly.

It is apparent that sample rate and nozzle size are closely coupled. The requirements for isokinetic or near-isokinetic nozzle flow sometimes impose a compromise on nozzle selection. The general order of priorities when choosing the sample rate is nozzle diameter (at least 1.4 mm), last stage jet velocity, and flow rate required for isokinetic sampling. Selection of nozzle diameter and impactor flow rate combinations for achieving near-isokinetic sampling conditions can be made from Figure C3.

If a choice must be made between undersized and oversized nozzles, undersized nozzles will usually result in lower sampling errors than will oversize.

### C.4.6 Use of Precollector Cyclones

In many instances the percentage (by weight) of material with sizes larger than the first impaction stage cut point is quite high. In such cases a precollector cyclone is necessary to prevent the upper impactor stages from overloading. A precutter should always be used for the first test. If the weight of material obtained by the precutter is greater than or equal to that on the first stage, the precollector should be used in all subsequent runs. Cyclones can be obtained from the impactor manufacturer or can be shop made. The use of two first stages in series has also been suggested and appears to be a valid approach; however, no data are available.

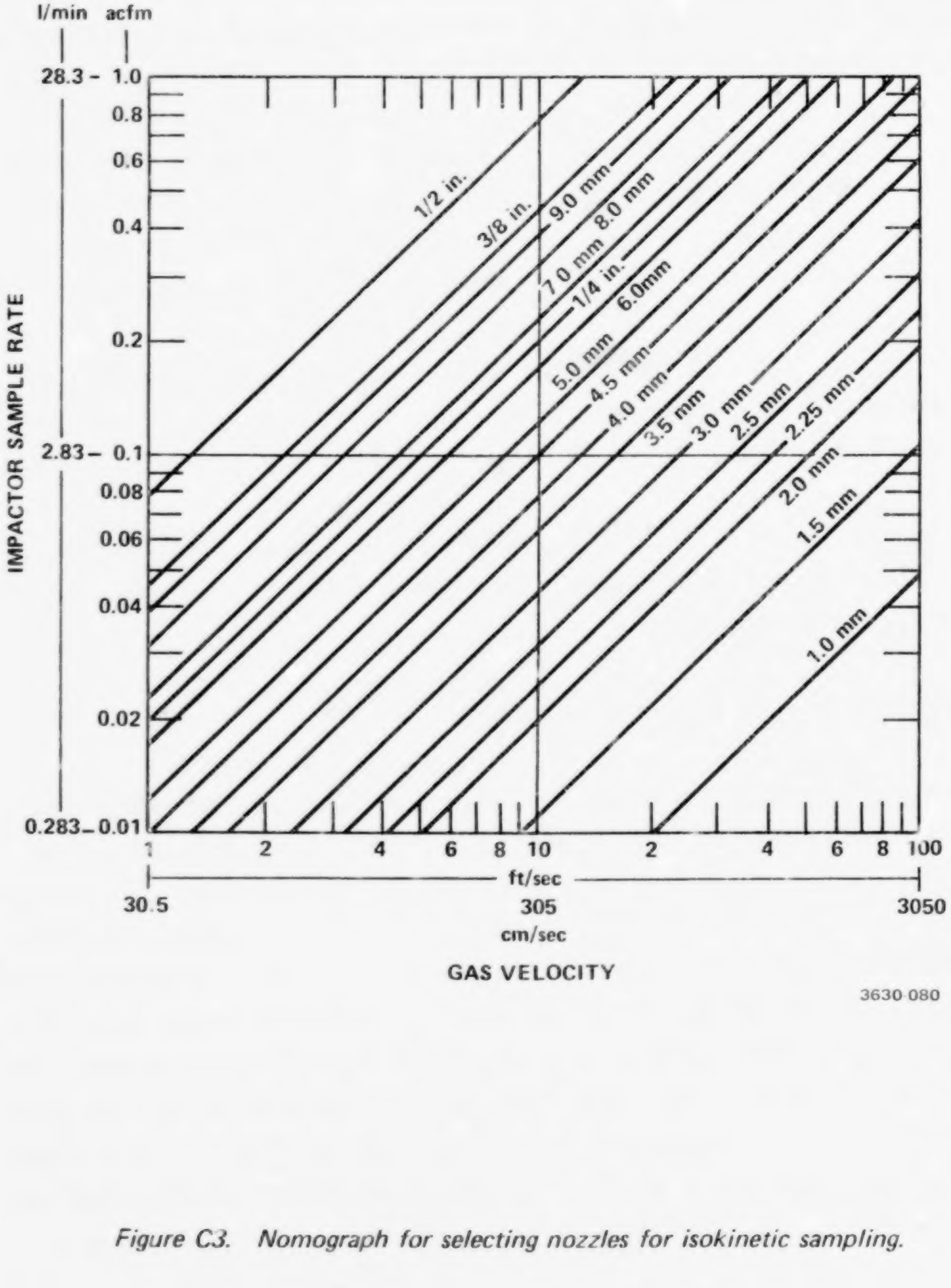


*Figure C3. Nomograph for selecting nozzles for isokinetic sampling.*

### C.4.7 Impactor Flow Rate Measurement

The flow rate through an impactor must be accurately measured in order to set the isokinetic sampling rate and to determine the correct impactor stage cut points. Unfortunately, it is usually very inconvenient, and sometimes impossible, to measure the impactor flow rate at the conditions present in the impactor. The gas is normally drier, cooler, and at a lower pressure by the time the flow rate is measured; and the flow must be corrected to impactor conditions. The use of calibrated orifices and dry gas meters is discussed below.

Units -- The equations presented are valid only if the units of the various terms are consistent. For instance, the pressure drop terms could be in units of mm $H_2O$ or cm $H_2O$ or something else, but all pressure drop terms must have the same units. The same is true for the other properties. Note that pressure and temperature are both absolute measurements.

Orifice meters -- The gas flow rate through a particulate orifice meter is related to the pressure drop across that orifice by an equation of the form:

$$Q^2 = C \frac{\Delta P}{\rho} \tag{C1}$$

where $Q$ = volumetric flow rate at upstream conditions,
$\Delta P$ = pressure drop across orifice,
$\rho$ = density of gas at upstream conditions, and
$C$ = dimensional constant, $(\text{length})^5(\text{mass})(\text{time})^{-2}(\text{force})^{-1}$.

Solving for the constant, C, in equation (C1), one obtains:

$$C = \frac{Q^2\rho}{\Delta P} \tag{C2}$$

As C is a constant at all conditions, its value can be obtained at a convenient set of conditions with a known flow rate and used later to calculate the actual sampling flow rate. Equation (C2) can be rewritten:

$$C = \frac{Q_c^{\,2}\ \rho_c}{\Delta P_c} \qquad \text{(C2a)}$$

The subscript "c" indicates that these parameters were determined during a calibration. Density and flow rate are at upstream conditions.

Substituting equation (C2a) into equation (C1) yields an equation suitable for obtaining flow rates from a calibrated orifice:

$$Q_m^{\,2} = \left[\frac{Q_c^{\,2}\ \rho_c}{\Delta P_c}\right] \frac{\Delta P_m}{\rho_m} \qquad \text{(C3)}$$

The subscript "m" denotes the parameters of the gas as it is being "measured". All are at conditions immediately upstream of the orifice.

For use with impactors, the measured flow rate, $Q_m$, must be converted to a flow rate at stack conditions, $Q_s$. Assuming that the stack gas was dried as well as altered in temperature and pressure, the stack flow rate is related to the measured flow rate by:

$$Q_s \left(1 - B_{wo}\right) \frac{P_s}{T_s} = Q_m \frac{P_m}{T_m} \qquad \text{(C4)}$$

where $B_{wo}$ = water removed from flue gas, expressed as a volumetric fraction,

$P$ = absolute pressure, and

$T$ = absolute temperature.

The subscript "s" refers to stack conditions.

At the usual conditions of relatively high temperature and low pressure which occur during stack sampling, the flue gas behaves very much like an ideal gas. The density of an ideal gas can be approximated as:

$$\rho = \frac{P(MW)}{(R)(T)} \qquad \text{(C5)}$$

where $R$ = the universal gas constant, and

$MW$ = the molecular weight of the gas.

Equations (C3), (C4), and C5) can be combined and rearranged into a form which gives the pressure drop which must exist across the calibrated orifice, $P_m$, to obtain the required impactor flow rate, $Q_s$.

$$\Delta P_m = \Delta P_c \frac{Q_s{}^2}{Q_c{}^2} \left(1-B_{wo}\right)^2 \left(\frac{P_s{}^2}{P_m P_c}\right) \left(\frac{T_m T_c}{T_s{}^2}\right) \left(\frac{MW_m}{MW_c}\right) \qquad \text{(C6)}$$

where $(MW)_m$ = molecular weight of the stack gas at the orifice, normally the dry molecular weight,

$(MW)_c$ = molecular weight of the calibration gas.

Dry gas meter -- The dry gas meter, like the orifice, can only directly measure the flow rate of the gas which passes through it. This measured flow rate can be converted to the flow rate through the impactor (which is at stack conditions) using equation (C4a):

$$Q_m = Q_s \left(\frac{T_m}{T_s}\right)\left(\frac{P_s}{P_m}\right)\left(1 - B_{wo}\right) \qquad \text{(C4a)}$$

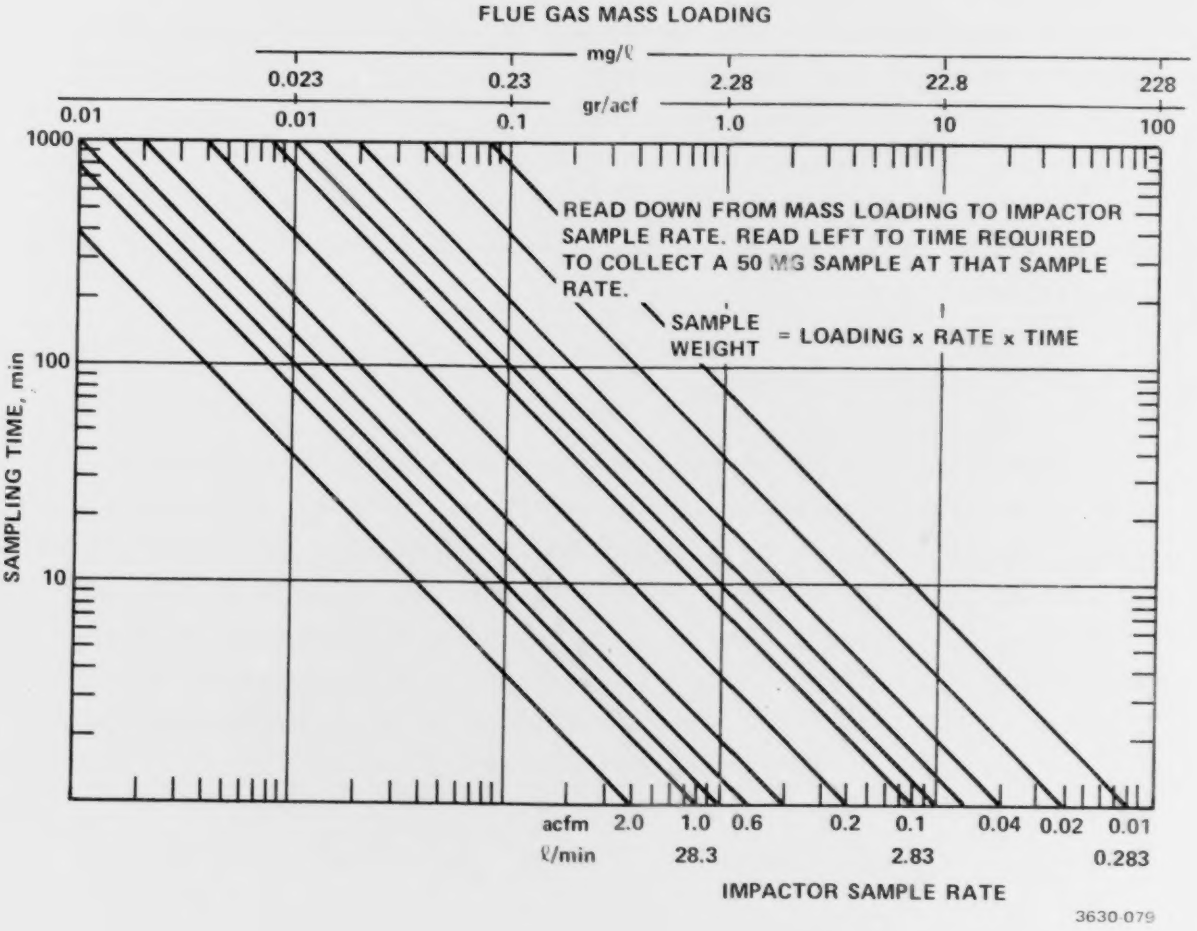
### C.4.8 Sampling Time

The length of the sampling time is dictated by mass loading and the particle size distribution. An estimate for initial tests can be obtained from Figure C4. Two conflicting criteria complicate the choice of the sampling time. It is desirable from the standpoint of minimizing weighing errors to collect several milligrams on each stage. However, most size distributions are such that the upper stages are overloaded and are re-entraining particles by the time the lower stages reach a few milligrams. A rule of thumb is that no stage should be loaded above 10 mg, but the determining factor is whether or not re-entrainment occurs. As is discussed later, a comparison of the relative distribution determined by a long run with that from a shorter (about half as long) run can be used to check on re-entrainment due to stage overloading.

### C.4.9 Readying the Impactor

As equipment is not always cleaned as well as it should be, the impactor should be inspected prior to use. The nozzles must be clean, gaskets in good shape, and the interior clean. Nozzles can be cleaned with fine wire if necessary.

After inspection, the impactor should be carefully loaded with the preweighed stage substrates and assembled. Teflon thread sealant tape or antiseize compound should be applied to the threads, especially when high temperatures (>215°C) are encountered. The thread sealant tape generally works better and causes fewer problems but probably cannot be successfully used at temperatures above 290°C.



READ DOWN FROM MASS LOADING TO IMPACTOR SAMPLE RATE. READ LEFT TO TIME REQUIRED TO COLLECT A 50 MG SAMPLE AT THAT SAMPLE RATE.

SAMPLE WEIGHT = LOADING x RATE x TIME

*Figure C4. Nomograph for sampling time selection (50 mg sample).*

If supplemental heating is required, a heating device and temperature monitor need to be added. A thermocouple mounted in the gas flow immediately after the impactor is best for controlling impactor cut points.

The supplemental heat can be supplied with either a heating mantle which has been made to fit the impactor or by using heating tapes. If the tapes are to be used, a heating tape of sufficient wattage is wrapped around the impactor. Glass fiber tape works well for holding the heating tape. Insulation such as asbestos tape is then wound around the impactor. Glass fiber tape again is used to hold the asbestos in place and also acts as additional insulation. The impactor can now be mounted on the appropriate probe, taken to the sampling position, and installed in the sampling system.

### C.4.10 Pre-Sample Checks

Impactors are prone to leak, and they must be checked at operating temperature for leaks. This can be done in several ways. The nozzle can be plugged and the impactor pressure-tested or vacuum-tested. Because impactors are basically a series of orifices, they should have a constant flow to pressure drop relationship. Checking the pressure drop on various flows of filtered air will point out deviations from normal operations--both leaks (external or internal) and plugged jets.

### C.4.11 Taking the Sample

The impactor should be preheated for at least 45 minutes before sampling. If supplemental heat is being used, the impactor should be brought up to temperature outside the duct and then allowed some time to equilibrate after insertion. The nozzle should not point into the flow field during this phase. Without supplemental heat, the whole warm-up is conducted within the duct, again with the nozzle pointed away from the flow field.

A predetermined flow rate must be maintained to ensure stable cut points. Any attempt to modulate flow to provide isokinetic sampling will destroy the utility of the data by changing the cut points of the individual stages. Rapid establishment of the correct flow rate is especially important for the short sampling times typically found at the inlets to control devices. Capping the nozzle during preheat in the flue is also desirable.

### C.4.12 Number of Sample Points

As the velocity and particulate distributions in industrial ductwork are unlikely to be ideal, a large number of samples are often required for accurate particulate measurements. A velocity traverse should be run to check on the velocity distribution. At least two points within a duct should be sampled in each measurement plane, and at least two samples taken at each of these points. These are the minimum sampling efforts and are appropriate only for locations with well developed flow profiles in the absence of significant concentration stratification. If the flow profile at the inlet or outlet is uncertain due to duct configuration and/or the mass loading is not uniform, the number of samples may need to be increased for reliable results.

## C.5 SAMPLE RETRIEVAL AND WEIGHING

### C.5.1 Impactor Clean-up

The careful disassembly of the impactor and removal of the collected particulate are essential to the success of the test program. The crucial points are to make sure that the collected material stays where it originally impacted and to remove all the particulate. After the sampling run, the impactor should be carefully removed from the duct without jarring it, removed from the probe, and allowed to cool. Disassembly can be difficult in some cases, particularly if the impactor was used at elevated temperatures.

Typically, not all of the particulate which collects in an impactor collects on the substrates. Some accumulates on the interior surfaces, especially in the nozzle. By convention, all of the particles collected upstream of a given impaction stage are assigned to that stage.

The collection of these "misdirected" particles is often troublesome. If the particles are hard and dry, they can be brushed off into the weighing container. A No. 7 Portrait brush or its equivalent is suggested, and care must be taken to prevent brush hairs from contaminating the sample. If the particles are sticky or wet, some type of washdown procedure should be used. The solvent must be considerably more volatile then the particulate matter.

### C.5.2 Drying and Weighing

All of the samples must be dried to constant weight, with 2 hour checks used to establish the uniformity of the weights. Hard, nonvolatile particles are often dried in a convection oven to 100°C, desiccated until cooled to room temperature, weighed, then check weighed. Volatile materials will require some other technique using low temperature. Whatever the technique used, constant weight of the sample with further drying is the criteria to be met.

### C.5.3 Data Logging

Permanent records should be kept of all pertinent information. It is generally necessary to keep records in three places--in the lab with the balance using a bound notebook, and using either looseleaf data forms of a bound notebook at both the inlet and outlet of the control device. Table C2 presents a fairly complete listing of the information required concerning an impactor run. In addition, records of the weighing of the catches must be kept. Notes should be taken on any abnormalities which occur and on the apparent condition of the stage catches.

## C.6 QUALITY ASSURANCE

The field use of cascade impactors is a difficult task. The accuracy required is more appropriate for a laboratory program than for a field test. There are many places in the operational sequence where errors can occur in spite of a conscientious effort to do a good job. Quality assurance attempts to discover inaccuracies before they are propagated throughout the test program. The techniques presented in this section are not the only ways to ensure quality data. However, they have been used successfully in field testing with impactors.

### C.6.1 Impactor Techniques

Glass fiber substrates -- As has been discussed previously, glass fiber substrates are not without problems. Two potentially serious problems are $SO_2$ uptake on the substrate and mechanical or manual abrasion of the filter mat.

TABLE C2. SAMPLING INFORMATION REQUIRED

Date
Time
Run Code Number
Impactor Type and Identification Number
Operator
Port Number/Sampling Location
Ambient Temperature
Ambient Pressure

Impactor In-Stack or Out-of-Stack
Impactor Orientation
Number of Traverse Points
Stack Pressure
Stack Temperature
Nozzle Diameter/Type
Probe Depth, if used
Stack Pitot Tube Delta P/Stack Gas Velocity

Desired Impactor Flow Rate for Isokinetic Sampling
Metering Orifice Identification Number
Metering Orifice Delta P
Impactor Temperature
Scalping Cyclone in Use? Identification
Prefilter Identification
Postfilter Identification
Substrate Set Identification
Pressure Drop Across Impactor

Test Start/End Time: Duration of Test
Gas Meter Start/End Readings? Gas Meter Volume
Agreement Between Meter and Orifice
Volume of Condensible $H_2O$ in Flue Gas
Gas Meter Temperature

The problem of $SO_2$ uptake on the substrate is discussed by Felix et al (1977). The two approaches available are to use a substrate which does not change weight in the flue gas or to precondition the substrate in filtered flue gas prior to weighing. Using a new glass fiber material, which does not react with the $SO_2$, may alter the particle retention characteristics of the impactor and change the impactor's calibration. This must be checked and the data reported. The use of preconditioned filter mats requires that the glass fiber substrates be preconditioned long enough to reduce the weight change during the expected duration of the impactor runs to 10 percent or less of the minimum stage weight. At the present time, this $SO_2$ reaction phenomenon is not well understood, and only rough guidelines are available. For some common glass fiber materials tested, the saturation times were on the order of 2 to 6 hours at the temperatures tested.

The applicability of the method chosen to overcome this substrate problem must be tested during the presurvey and periodically during the test runs by running blanks.

Glass fiber substrates must be handled carefully to prevent damage and possible loss of fibers. Loose surface fibers should be removed by shaking prior to initial weighing. After weighing, every precaution must be taken to prevent the loss of any part of the substrate. One approach which will quantify the problem of substrate abrasion is to prepare a substrate set, load the impactor, then disassemble and reweigh.

Greased metal substrates -- The problems which occur with the use of greased substrates are usually related to the properties of the grease. A grease which has been applied too heavily or has a low viscosity at operating temperature can be physically blown off the impactor stage. The grease could also react chemically with the flue gas or be excessively volatile at the operating temperature. Again, these phenomenon must be checked during the presurvey and periodically during the test program.

Re-entrainment -- Re-entrainment is the phenomenon of an impacted particle being blown off the stage on which it was collected downstream. This can be caused by excessive jet velocities or by overloaded stages. The effect of re-entrainment can be serious, because only a few large particles on a small particle stage will considerably affect the size distribution.

One way to spot re-entrainment is to very carefully examine the stage catches. If, for example, a low velocity through the jets resulted in a well-defined pile of particulate and a high velocity sample gave a diffuse deposit, re-entrainment should be suspected at the high sampling rate. Microscopic examination of the lower stages and final filter for large particles (which should have been collected upstream) is another way to check for re-entrainment.

Re-entrainment due to stage overloading can be detected by running two otherwise identical tests for two different test durations. If the two size distributions are not the same, overloading should be suspected at the higher stage loadings.

Impactor leaks -- Two types of leaks can occur with impactors--internal or external. A flow rate versus pressure drop check of a pressure test will pick up most leaks. An internal leak, where part of the airstream is bypassing the proper flow path, will give results similar to re-entrainment. Leak checks must be made at operating temperature.

General procedure -- A general procedure for impactor use, concentrating on quality assurance, has been outlined below:

1. Prepare Impactor
    a. Wash impactor. Use ultrasonic cleaner if available.
    b. Visually check cleanliness. Jets must be clear, sidewalls clean. Must be done in good lighting.
    c. Obtain preweighed substrates and assemble impactor.

2. Sampling

    a. Assemble impactor train and heat to operating temperature.
    b. Leak check the impactor.
    c. Sample with impactor.
    d. Disassemble impactor, examine stage catches and impactor walls. Note any anomalies.

3. Substrate and Re-entrainment Checks

    a. Check during presurvey.
    b. Check substrates if flue gas composition changes significantly.

### C.6.2 Weighing Techniques

Precision and calibration -- The manufacturer's directions should be followed when operating the balance. The balance should be calibrated at least once a day. The repeatability of measurements should be checked by repeatedly weighing a substrate and a test weight.

Technique -- The assembly and disassembly of an impactor should have no effect on the substrate weights. This should be checked by weighing up a set of substrates, assembling them in an impactor, then disassembling and reweighing. Any weight losses from this process should be within the repeatability of the balance (approximately 0.02 milligram for an electrobalance). Dry weight checks are made by desiccating the substrate, weighing, then desiccating again and reweighing. When the agreement is within the repeatability of the balance, dry weight has been achieved.

### C.6.3 General Notes

Spare parts -- The well equipped sampling team will travel with an adequate supply of spares. Improvisation due to an equipment failure can lead to poor quality data.

Flow meters -- At least two flow meters should be used in series. If they do not agree, the problem should be investigated.

Pumps -- Typically, vacuum pumps in sampling trains leak. For this reason, the flow meters should be upstream of the pump.

### C.6.4 Data Analysis

Final filter data -- The fine particulate information obtained from the final filter can sometimes be misleading. It is assumed for analysis that a stage captures everything larger than its $D_{50}$, and captures nothing smaller. A real stage misses some large particles. Under some conditions (including but not limited to re-entrainment), large particles will penetrate to the final filter. In this case the size distribution will be skewed towards the small particles. Microscopic examination of the final filter may provide an indication of this problem. If it occurs, the best choice in data analysis is probably to ignore the final filter on runs where this phenomenon was encountered.

Cumulative size data analysis -- If either a probe or a precutter cyclone is used with an impactor, the resultant probe losses and precutter catches must be included in cumulative size analysis. Failure to do so will lead to an incorrect cumulative distribution.

Inspection of data -- After the data have been collected, they should be examined for any inconsistencies and outliers should be rejected before the final averaging is done.

## C.7 USE OF COMMERCIAL IMPACTORS

### C.7.1 Brink BMS-11 Cascade Impactor

Additional information on the following impactors can be found in Section 2.3.2 under Table I.

The Brink impactor is a five-stage, low sample rate, cascade impactor, suitable for measurements in high mass loading situations. The Brink uses a single round jet on each of its stages.

Sampling rate -- The usual sampling rates for the Brink are in the range of 9-33 $cm^3/sec$ (0.02 to 0.07 acfm). The sampling rate must be low enough to prevent re-entrainment of particles from the lower stages. With hard, bouncy particulate, the last stage nozzle velocity must be less than 30-35 m/sec with ungreased collection plates and less than 60 m/sec with greased collection plates.

Collection substrates and adhesives -- The Brink impactor collection stage is too heavy to use without some type of substrate insert. Foil cups are commonly preformed and fitted into the collection cups of the Brink stages. If grease is to be used, the top stages require about 5 or 6 drops of solution while the bottom stages normally require only about one drop in the center of the cup. Glass fiber substrates cut to fit the collection cups have also been found satisfactory in many situations.

Back up filter -- The Brink back up filter is normally made of binderless glass fiber filter material. Two 2.5 cm diameter disks of filter material are placed under the spring in the last stage of the impactor. The filter is protected by a Teflon O-ring and the second filter disk acts as a support.

Precutter cyclone -- A precutter cyclone for the Brink is not commercially available.

Sampling train -- The Brink uses the usual type of sampling train. Orifices on the order of 0.77, 1.52, and 2.29 mm in diameter allow full coverage of its range of sampling rates at reasonable pressure drops.

Brink clean-up -- Careful disassembly of a Brink impactor is necessary for obtaining good stage weights. If a precollector cyclone has been used, all material from the nozzle to the outlet of the cyclone is included with the cyclone catch. All of this material should be brushed onto a small, tared, 2.5 x 2.5 cm aluminum foil square to be saved for weighing. Cleaning the nozzle is also important, especially if it is a small bore nozzle. All material between the cyclone outlet and the second stage nozzle is included with material collected on the first collection substrate. All appropriate walls should be brushed off, as well as around the underside of the nozzle, where as much as 30 percent of the sample has been found.

### C.7.2 Amdersen Mark III Stack Sampler

The Andersen impactor is a relatively high sample rate impactor. The normal sample rate is about 236 cm /sec (0.5 acfm). The Andersen is a multiple jet, round hole impactor.

Sampling rate -- The nominal Andersen sampling rate is given above. As with other cascade impactors, the flow rate must be low enough to prevent re-entrainment of impacted dust.

Collection substrates and adhesives -- Andersen substrates are obtained precut from the manufacturer. The substrates are glass fiber and of two types--one cut for the odd numbered stages, one for the even. As discussed earlier, normal Andersen substrates have a tendency to absorb $SO_2$ on basic sites in the substrate and therefore gain weight.

The Andersen requires careful assembly, as overtightening will cause the substrates to stick to the metal separator rings.

Back up filter -- The Andersen uses a 63.5 mm diameter disk placed above the final F-stage. (This F-stage is an option not normally included with the standard stack head.) The filter should be cut from binderless glass fiber filter material, such as Reeve-Angel 934AH filter paper.

Precutter cyclone -- A precutter cyclone for the Andersen is available from the manufacturer. It is necessary to have a 6-inch diameter or larger sampling port when using the precutter cyclone with its nozzle.

Andersen sampling train -- The Andersen requires the usual type of sample train. The pumping and metering systems of the commercial EPA Method 5 mass sampling trains are appropriately sized for use with the Andersen.

Care should be exercised never to allow a gas flow reversal to occur through the impactor. Material could be blown off the collection substrate onto the underside of the jet plate or the collection substrates could be disturbed. A check valve or maintenance of a very low flow while removing the impactor from the duct avoids this problem.

Andersen clean-up -- Cleaning an Andersen impactor is difficult. Foils should be cut to hold the substrates, and each foil and substrate weighed together before and after the run. For disassembly, the foil to hold the stage 1 substrate should be laid out. Next the nozzle and entrance cone should be brushed out and onto the foil. Then the material on stage 0 should be brushed onto the foil. The stage 1 filter substrate material should then be placed on the foil and, finally, the top of the stage 1 plate, O-ring, and cross piece should be brushed off. Depending on how tightly the impactor was assembled, some filter material may stick to the O-ring edge contacting the substrate. This should be carefully brushed onto the appropriate foil. This process is continued through the lower stages. Finally, the after filter is carefully removed.

### C.7.3 University of Washington Mark III (Pilat) Impactor

The Mark III impactor is a seven-stage, high flow rate device with generally the same characteristics as the Andersen. The Mark III is a round hole, multiple jet impactor.

Sample rate -- The Mark III sampling rate is on the order of 236 $cm^3$/sec (0.5 acfm). The flow rate must be low enough to keep scouring of impacted particles to a minimum.

Collection substrates and adhesives -- The Mark III has often been used with supplementary foil (aluminum or stainless steel) substrates. These substrates require the use of grease for easily re-entrained particles. Enough of the grease solution is placed evenly on the substrate to adequately cover the area under the jets. The normal cautions on the use of greased substrates apply as discussed in the text.

Precutter cyclone -- A BCURA (British Coal Utilization Research Association) designed precutter cyclone is available from the manufacturer.

Mark III sampling train -- As the Mark III is a high flow rate device, its sampling train is similar to that of the Andersen.

Mark III clean-up -- Mark III impactor clean-up is similar to that for the Brink. Some problems have been noted with O-rings sticking rather tenaciously and care must be exercised not to dislodge the sample while trying to separate the stages.

### C.7.4 Meteorology Research, Inc. (MRI) Model 1502 Cascade Impactor

The MRI impactor is a high flow rate sampler. The body of the instrument is constructed from quick-disconnect rings which allow flexibility in configuration of the impactor and a positive gas seal between stages. The impactor uses multiple round jets in its stages.

Sampling rate -- The sampling rate is nominally 236 $cm^3$/sec (0.5 acfm) in the seven-stage configuration. Higher flow rates have been used by removing the last stages.

Collection substrates and adhesives -- The MRI collection disc is a self-supporting foil (316 stainless steel) which is functionally similar to the collection cup or tray and inserts used in other impactors. The collection discs are mass produced and normally are used only once and discarded.

Grease applied as described earlier is recommended for most applications.

Back-up filter -- The MRI impactor has a built-in filter holder for 47 mm diameter filters. Normally, binderless glass filters are used. Filter losses can be prevented by placing tared Teflon washers on both sides of the filter during the test.

MRI sampling train -- The MRI sampling train is similar to that of the Andersen.

MRI clean-up -- The clean up of the MRI impactor is similar to the Brink. The device is clamped in a vice and all of the sections and nozzles are loosened with wrenches. The wall losses are carefully brushed onto the appropriate collection disc. Care is taken not to brush contamination from the threads into the sample. A tared foil dish is used to hold the back up filter. Any worn O-rings should be replaced and the whole unit carefully cleaned before the next test.

### C.7.5 Sierra Model 226 Source Cascade Impactor

The Sierra impactor is a six-stage, high sample rate cascade impactor. The Sierra instrument uses a radial-slot design.

Sampling rate -- The Sierra impactor has a nominal sampling rate 236 $cm^3$/sec (0.5 acfm). The flow rate must be low enough to prevent re-entrainment of particles. Laboratory research (Cushing, 1976) has shown that a flow rate of 118 $cm^3$/sec (0.25 acfm) gives better stage collection efficiency characteristics for this impactor.

Collection substrates and adhesives -- Substrates for the Sierra are obtained precut from the manufacturer. These are glass fiber substrates and should be checked for weight gain. Stainless steel substrates are also available and these should normally be coated with grease as described earlier.

Back-up filter -- The back-up filter uses a 47 mm glass fiber filter mat. It is supported by a screen from below.

Precutter cyclone -- A precutter cyclone is available from the manufacturer.

Sampling train -- The sampling train for the Sierra is similar to that of the Andersen.

Clean-up -- Clean-up of the Sierra is fairly similar to Andersen clean-up. Care should be taken to be sure the glass fiber substrates are removed intact.

## C.8 DATA ANALYSIS

### C.8.1 General Discussion

The majority of impactor data reduction is done using a technique referred to as the "$D_{50}$" method. In this method it is assumed that all of the particles caught by an impactor stage consist of particles having diameters equal to or greater than the $D_{50}$ of that stage, but less than the diameter of the stage above. The mass caught by the first stage or cyclone is considered to be equal to or greater than the $D_{50}$ of that stage, but smaller than the largest particle diameter present in the aerosol. The largest diameter may be determined, approximately by microscopic examination of the stage catch, or it may be assigned some reasonable arbitrary value, say 100 μm.

Particle size distributions may be presented on a differential or a cumulative basis. When using the $D_{50}$ method, either type of presentation may be easily employed.

The size parameter reported can be aerodynamic diameter, aerodynamic impaction diameter, or Stokes diameter. In all cases, the particles are assumed to be spherical. The method of reporting diameters depends to a large extent upon the ultimate use of the size distribution information. For this reason it is suggested that the data be reported in three parallel sets: one set based on aerodynamic impaction diameter, one based on aerodynamic diameter, and one based on the Stokes diameter.

### C.8.2 Calculation of Impactor Stage $D_{50}$'s

The reduction of field data obtained with a cascade impactor can sometimes be troublesome and time consuming because of the computations involved. The equations below are based on the motion of particles in the Stokes regime for which the Reynolds number is less than 1000. Although this is not always true for impactors, the equations are often a good approximation. The basic equation that defines the theoretical impaction behavior of a given stage of a cascade impactor is

$$\psi_j = \frac{\rho_p d_p{}^2 V_j C}{18\ \mu D_j} \tag{C7}$$

where $\psi_j$ is the inertial impaction parameter,
$\rho_p$ is the particle density, gm/cm³,
$V_j$ is the velocity of the gas (and particles) in the impactor jet (cm/sec),
$\mu$ is the gas viscosity, poise,
$D_j$ is the diameter (width for slots) of the jet, cm,
$C$ is the slip correction factor, and
$d_p$ is the particle diameter, cm.

If the value of $\psi$ for 50 percent collection, $\psi_{50}$, can be determined, equation C7 can be inverted to give the stage $D_{50}$ for a wide range of test conditions. Historically, the experimental values reported by Ranz and Wong (1952) have been used. These are:

For round jets, $\psi_{50}$ = 0.145, and
For rectangular jets, $\psi_{50}$ = 0.44.

Subsequent studies, however, have shown that there is no universal value for $\psi_{50}$ and the actual value must be determined by calibration for each impactor design. Several papers and reports are available which tabulate stage constants of different impactors, and outline procedures for impactor calibration, Harris (1977) and Cushing (1976).

From equation C7,

$$D_{50_j} = \left(\frac{18\psi_{50_j}\mu D_j}{C\rho_p V_j}\right)^{\frac{1}{2}}. \tag{C8}$$

As equation (C8) is written, with the actual particle density and the calculated slip correction factor, it defines the Stokes diameter. If the particles are treated as if their density, $\rho_p$, was 1.0, equation (C8) defines the aerodynamic diameter. If the slip correction factor is also assumed to be equal to 1.0, the aerodynamic impaction diameter is defined by equation (C8). See Section A.3.6.

Since C, the slip correction factor, contains D, this equation must be solved by iteration where $D_{50}$ and C are calculated alternately.

Equation C8 may be written more conveniently in terms of the test parameters: For round jet impactors

$$D_{50_j} = \left(\frac{14.1\ \psi_{50_j}\ \mu D_j{}^3 X_j P_j}{C\rho_p Q_s P_s}\right)^{\frac{1}{2}}, \tag{C9}$$

where $X_j$ is the number of jets on the stage,
$P_j$ is the absolute pressure downstream of the jet(s), mm Hg,
$P_s$ is the absolute pressure in the stack, mm Hg, and
$Q_s$ is the sample flow rate, cm$^3$/sec.

and for rectangular jet impactors

$$D_{50_j} = \left( \frac{18 \psi_{50_j} \mu W_j^{\,2} L_j P_j}{C \rho_p Q_s P_s} \right)^{\frac{1}{2}} \qquad \text{(C10)}$$

where $W_j$ is the jet width, cm, and
$L_j$ is the total jet length, cm.

One approach that can be used to simplify the computations is to develop curves for the impactor stage cut points at one set of conditions--e.g., air at standard conditions and a particle density of 1.0. Then a suitable correction factor can be applied to these curves for the actual sampling conditions. Unfortunately, further steps are involved in making the correction factor simple enough to be of value. Therefore, the use of this type of approach suffers from some restrictions.

All of the assumptions and calculations involved in going from equations C9 to C10 to the calibration curve can be quite awkward, particularly in cases where different types of sources are being sampled. The best and easiest approach is to write or obtain a computer program based on the rigorous equations given initially. Such a program can calculate impactor stage cut points, compute concentrations of particles in each size range, determine the baghouse efficiency, and plot graphs.

A sophisticated computer data reduction program is available from the EPA (Johnson, et. al., 1978) and also less powerful programs are available for the Hewlett Packard HP-65 (Ragland, 1976) and HP-25 (Ragland, 1977) programmable calculators.

### C.8.3 Cumulative Particle Size Distributions

Impactor data may be presented on a cumulative basis by summing the mass on all the collection stages and back up filter, and plotting the fraction of the mass below a given size versus size. This is frequently done on special log-probability paper. Semi-log paper may be preferable for distributions that are not log-normal.

Cumulative distributions are very easy to understand and present the data with clarity. Cumulative distributions, however, have a couple of disadvantages when compared to differential distributions. An error in stage weight will be propagated throughout a cumulative analysis, but will be isolated by the differential approach. Also the differential method does not involve the use of total mass concentration or total size distribution from diameters of zero to infinity, and so is useful in comparing instruments with overlapping but different size fractionation ranges and different stage cut points while cumulative analysis is not.

When cumulative plots are used, the abscissa is normally the logarithm of the particle diameter and the ordinate is the weight percent smaller than this size. The value of the ordinate at a given $(D_{50})_k$ would be

$$\text{Weight percent smaller than } (D_{50})_k = \frac{\sum_{i=o}^{k-1} \Delta M_i}{\sum_{i=o}^{K} \Delta M_i} \times 100\%, \qquad \text{(C11)}$$

where i = o corresponds to the filter,
i = k corresponds to the stage under study, and
i = K corresponds to the coarsest jet or cyclone.

This equation requires that the stages be counted from the final filter up. There is no $(D_{50})_o$, as the "o" corresponds to the filter. $(D_{50})_1$ is the cut point of the last stage, which collects mass, $\Delta M_1$.

### C.8.4 Differential Size Distributions

Differential particle size distributions are used to plot the relative concentration versus particle diameter. The area under the frequency curve, between two designated diameters, is equal to the mass of particles in that size range. Differential curves may be obtained directly from the reduced impactor data, or by differentiation of curves fitted to the cumulative particle size distributions.

Many cascade impactors are designed so that the relationship between successive stage $D_{50}$'s is logarithmic. For this reason, and to minimize graphical scaling problems, the differential particle size distributions are plotted on log-log or semi-log paper with $\Delta M/\Delta(\log D)$ as the ordinate and geometric mean of $\Delta(\log D)$ as the abscissa. The mass on stage "n" is designated by $M_n$ and is, in approximation, the particulate mass with diameters between

$(D_{50})_n$ and $(D_{50})_{n+1}$. The $\Delta(\log D)$ associated with $\Delta M_n$ is $\log(D_{50})_{n+1} - \log(D_{50})_n$. Using these approximations, the derivative term (ordinate) associated with stage "n" is:

$$\Delta M/\Delta(\log D)|_n \quad \frac{\Delta M_n}{(\log D_{50})|_n} = \frac{\text{Mass on Stage "n"}}{\log(D_{50})_{n+1} - \log(D_{50})_n}, \qquad \text{(C12)}$$

and the abscissa, $D_{geo}$, is

$$\left[(D_{50})_{n+1} \times (D_{50})_n\right]^{\frac{1}{2}} \qquad \text{(C13)}$$

Usually, smooth curves are fitted through these computed points for convenience in averaging the data and calculating the baghouse efficiency. As many tests as possible should be obtained for each fabric filter operating condition and the results averaged after outliers have been identified and discarded.

The fractional penetration of the fabric filter is determined by dividing the magnitude of the outlet $\Delta M/\Delta(\log D)$ plot by the magnitude of the inlet at several corresponding values of $D_{geo}$. The efficiency is equal to the penetration subtracted from unity, multiplied by 100%. Both the efficiency and penetration can be conveniently plotted on a single log-probability graph.

### C.8.5 Confidence Limits of Reduced Data

After data from the individual cascade impactor runs have been reduced, then averages for these inlet and outlet tests can be calculated. Generally the data are presented as (1) Cumulative Mass Versus Particle Size, (2) Cumulative Per Cent Mass Versus Particle Size, and (3) $\Delta M/\Delta(\log D)$ Versus Geometric Mean Particle Size. To calculate the Upper and Lower 90% Confidence Limits for the data, the following equations are used:

$$UCL_{90} = \text{Average} + \frac{t_{90}\sigma}{\sqrt{N}} = \text{Average} + C.I._{90} \qquad (C14)$$

$$LCL_{90} = \text{Average} - \frac{t_{90}\sigma}{\sqrt{N}} = \text{Average} - C.I._{90} \qquad (C15)$$

where Average = The average of the N values for $\Delta M/\Delta \log D$ or $\Delta N/\Delta \log D$ at a particular particle size.

$t_{90}$ = The Student's t distribution for 90% confidence limits with N sets of data.

$\sigma$ = The Standard Deviation of the N sets of data.

N = The number of data values in the average.

The average cumulative mass and cumulative per cent graphs are obtained from $\Delta M/\Delta \log D$ data by piecewise integration, after discarding outliers. The confidence interval of the cumulative graph, at a particular size, is equal to the square root of the sum of the squares of the confidence intervals of each size increment of the differential graph less than or equal to that size.

Clearly, the average values are more reliable and the confidence limits are small when the number of data points is large.

A determination of fabric filter fractional penetration is made by taking the ratio of the average $\Delta M/\Delta \log D$ outlet values to the average $\Delta M/\Delta \log D$ inlet values at a series of particle sizes. The confidence limits of the calculated penetration are given by:

$$UCL_{90} = \text{Average Penetration} + CIP_{90} = P + CIP_{90} \qquad (C16)$$

and

$$LCL_{90} = \text{Average Penetration} - CIP_{90} = P - CIP_{90} \qquad (C17)$$

where:

$$CIP_{90} = \left\{ \frac{(C.I._{90},\ \text{Outlet})^2}{(\text{Inlet Average})^2} + P^2 \frac{(C.I._{90},\ \text{Inlet})^2}{(\text{Inlet Average})^2} \right\}^{\frac{1}{2}} \qquad \text{(C18)}$$

and

$$P = \frac{\text{Outlet Average}}{\text{Inlet Average}} .$$

### C.8.6 Cascade Impactor Data Reduction--Sample Calculation

This section contains a detailed description of the calculations that are required to derive particle size distributions and a fabric filter fractional efficiency curve from raw impactor data. The specific example given is for a single hypothetical test performed with an Andersen impactor. The calculation procedures outlined here can be used with any impactor, however. Normally the results of all tests made under the same process and baghouse operating conditions are grouped and averaged, and confidence limits calculated as described in Section C.8.5.

This discussion is based on Table C3 which was generated by a computer program (Johnson, et. al., 1978). All of the calculations, however, can be done on programmable calculators. In the example shown, the data is reduced using a particle density of 1.35 gm/cm$^3$; thus the diameters reported are Stokes diameters. For aerodynamic, or aerodynamic impaction diameters, ρ or ρ and $C_{\rho}$, are set to unity, respectively. (ρ is particle density and C is the slip correction factor.)

Information obtained from the data log sheets for each test is printed at the top of Table C3. The maximum particle diameter is measured by examining the particles collected on the first stage (or first cyclone) with an optical microscope. Gas analysis

# TABLE C3

## SAMPLE CALCULATION - INPUT DATA AND RESULTS

Hypothetical Andersen

Impactor Flowrate = 0.500 ACFM | Impactor Temperature = 400.0 F = 204.4 C | Sampling Duration = 20.00 Min

Impactor Pressure Drop = 1.5 In. of Hg | Stack Temperature = 400.0 F = 204.4 C

Assumed Particle Density = 1.35 gm/cu.cm. | Stack Pressure = 26.50 In. of Hg | Max. Particle Diameter = 100.0 Micrometers

Gas Composition (Percent) | CO2 = 0.95 | CO = 0.00 | N2 = 76.53 | O2 = 20.53 | H2O = 1.00

Calc. Mass Loading = 8.0711E -03 gr/acf | 1.4948E -02 gr/dscf | 1.8470E+01 mg/acm | 3.4207E+01 mg/dscm

| Impactor Stage | S1 | S2 | S3 | S4 | S5 | S6 | S7 | S8 | FILTER |
|---|---|---|---|---|---|---|---|---|---|
| Stage Index Number | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
| D50 (Micrometers) | 10.74 | 9.95 | 6.36 | 4.19 | 2.22 | 1.29 | 0.69 | 0.33 | |
| Mass (Milligrams) | 0.72 | 0.50 | 0.53 | 0.09 | 0.38 | 1.43 | 1.25 | 0.04 | 0.39 |
| MG/DSCM/STAGE | 4.71E+00 | 2.62E+00 | 3.47E+00 | 5.89E-01 | 2.49E+00 | 9.35E+00 | 8.18E+00 | 2.62E+01 | 2.55E+00 |
| Cum. Percent Of Mass Smaller Than D50 | 86.24 | 78.59 | 68.46 | 66.74 | 59.47 | 32.13 | 8.23 | 7.46 | |
| Cum. (MG/ACM) Smaller Than D50 | 1.59E+01 | 1.45E+01 | 1.26E+01 | 1.23E+01 | 1.10E+01 | 5.93E+00 | 1.52E+00 | 1.38E+00 | |
| Cum. (GR/ACF) Smaller Than D50 | 6.96E-03 | 6.34E-03 | 5.53E-03 | 5.39E-03 | 4.80E-03 | 2.59E-03 | 6.64E-04 | 6.02E-04 | |
| Cum. (GR/DSCF) Smaller Than D50 | 1.29E-02 | 1.17E-02 | 1.02E-02 | 9.98E-03 | 8.89E-03 | 4.80E-03 | 1.23E-03 | 1.12E-03 | |
| Geo. Mean Dia. (Micrometers) | 3.28E+01 | 1.03E+01 | 7.96E+00 | 5.17E+00 | 3.05E+00 | 1.69E+00 | 9.43E-01 | 4.74E-01 | 2.31E-01 |
| DM/DLOGD (MG/DSCM) | 4.86E+00 | 7.94E+01 | 1.79E+01 | 3.25E+00 | 8.99E+00 | 3.99E+01 | 2.98E+01 | 8.09E-01 | 8.47E+00 |
| DN/DLOGD (NO. PARTICLES/DSCM) | 1.95E+05 | 1.02E+08 | 5.01E+07 | 3.33E+07 | 4.48E+08 | 1.16E+10 | 5.03E+10 | 1.08E+10 | 9.74E+11 |

Normal or standard conditions are 21°C and 760 mm Hg samples are taken at the same time the impactor is run. The mass loading is calculated from the total mass of the particles collected by the impactor, and listed in four different systems of units after the heading CALC. MASS LOADING. The symbols are defined as:

GR/ACF - grains per actual cubic foot of gas at stack conditions of temperature, pressure, and water content.

GR/DSCF - grains per dry standard cubic foot of gas at engineering standard conditions of the gas. Engineering standard conditions are defined as 0% water content, 70°F, and 29.92 inches of Hg.

MG/ACM - milligrams per actual cubic meter of gas at stack conditions of temperature, pressure, and water content.

MG/DSCM - milligrams per dry standard cubic meter of gas at engineering standard conditions of the gas. Engineering standard conditions are defined as 0% water content, 21°C, and 760 mm of Hg (Torr).

The conditions at which the impactor was run determine stage $D_{50}$ cut points. These are calculated by iterative solution of the following equations:

$$D_{50_i} = \left[\frac{14.1\ \mu D_{c_i}{}^3 \psi_{50_i} P_{s_i} X_i}{\rho_p\ Q\ P_0\ C_i}\right]^{1/2} \tag{C19}$$

$$C_i = 1 + \frac{2\ \lambda_i}{D_{50_i} \times 10^{-4}}\left[1.23 + 0.41\ \mathrm{EXP}\left(-0.44\ \frac{D_{50_i}}{\lambda_i} \times 10^{-4}\right)\right] \tag{C20}$$

where $D_{50_i}$ = stage (i) cut point (cm),
$\mu$ = gas viscosity (poise)
$D_{c_i}$ = stage jet diameter (cm),

$P_{s_i}$ = local pressure at stage jet (mm Hg),

$\rho_p$ = particle density ($gm/cm^3$),

$Q$ = impactor flow rate ($cm^3/sec$),

$P_0$ = ambient pressure at impactor inlet (mm Hg),

$C_i$ = Slip Correction Factor,

$\lambda_i$ = gas mean free path (cm),

$x_i$ = number of holes per stage (i), and

$\psi_{50_i}$ = inertial impaction parameter of stage (i).

To find the pressure $P_{s_i}$ at each impactor stage, the following equation is used:

$$P_{s_i} = P_0 - \left(F_i\right) (DP), \tag{C21}$$

where $P_0$ is the ambient pressure at the impactor inlet,

$F_i$ is the fraction of the total impactor pressure drop at each stage, and

DP is the total pressure drop across the impactor.

The total pressure drop DP across the impactor is given by the following equation.

$$DP = K\, Q^2\, \rho\, MM, \tag{C22}$$

where K = Empirically determined constant for each impactor,

Q = Flow rate through impactor ($cm^3/sec$),

$\rho$ = Gas density (gm/cm ), and

MM = Mean Molecular Weight of gas (gm/gm-mole).

To calculate the gas mean free path, $\lambda_i$, for each impactor stage, the following equation is used:

$$\lambda_i = \frac{2\mu}{1.013 \times 10^6 P_{s_i}} \times \sqrt{\frac{8.31 \times 10^7 T_K}{3 MM}} \qquad (C23)$$

where $\mu$ is the gas viscosity (poise),

$P_{s_i}$ is the pressure at each impactor stage (atm),

$T_K$ is the gas temperature at the impactor stage (°K), and
MM is the average flue gas molecular weight.

To find the viscosity of the flue gas, $\mu$, the viscosity of the pure gas components of the flue gas must first be found. Viscosity is a function of temperature, and the temperature difference in different flue gases can be quite significant. The following equations (derived from curves fitted to viscosity data from the Handbook of Chemistry and Physics, Chemical Rubber Company Publisher, 54 Edition, 1973-1974, pp. F52-55), are used to find the viscosities of $CO_2(\mu_1)$, $CO(\mu_2)$, $N_2(\mu_3)$, $O_2(\mu_4)$ and $H_2O(\mu_5)$.

$\mu_1 = 138.494 + 0.499T - 0.267 \times 10^{-3}T^2 + 0.972 \times 10^{-7}T^3$
$\mu_2 = 165.763 + 0.442T - 0.213 \times 10^{-3}T^2$
$\mu_3 = 167.086 + 0.417T + 0.417 - 0.139 \times 10^{-3}T^2$
$\mu_4 = 190.187 + 0.558T - 0.336 \times 10^{-3}T^2 + 0.139 \times 10^{-6}T^3$
$\mu_5 = 87.800 + 0.3$[illegible]$ + 0.238 \times 10^{-4}T^2$

where T is the temperature of the flue gas in degrees Celsius. The units of $\mu$ are $10^{-6}$ g/cm-sec. Next, these values of $\mu_1$ through $\mu_5$ are used in a general viscosity equation for a mixture of any number of components (See "A Viscosity Equation for Gas Mixtures" by C. R. Wilke, Journal of Chemical Physics, Volume 8, Number 4, April 1950, page 517) used to find the viscosity of the flue gas:

$$\mu = \sum_{i=1}^{n} \frac{\mu_i}{\left[1 + \frac{1}{X_i} \sum_{\substack{j=1 \\ j \neq i}}^{j=n} X_j \phi_{ij}\right]}$$

where $\phi_{ij}$ is given by the equation:

$$\phi_{ij} = \frac{\left[1 + \mu_i/\mu_j \,^{\frac{1}{2}} \; M_j/M_i \,^{\frac{1}{4}}\right]^2}{4/\ 2\left[1 + \left(M_i/M_j\right)\right]^{\frac{1}{2}}} \tag{C25}$$

and M = molecular weight of a component in the mixture,

X = mole fraction of a component in the mixture,

$\mu$ = viscosity, gm/cm-sec; $\mu_1$, $\mu_2$, etc. refer to the pure components at the temperature and pressure of the mixture, $\mu$ is the viscosity of the mixture, and

$\phi$ = dimensionless constant defined above.

Below these data the information pertinent to each stage is summarized in columnar form in order of decreasing particle size from left to right. Thus S1 is the first stage, S8 is the last stage, and FILTER is the back-up filter. If a cyclone was used, then to the left of S1 a column labelled CYC would appear and information relevant to the cyclone would be listed in this column. Beneath each impactor stage number is listed the corresponding stage index numbers, which also serve as identification for the stages. Directly beneath these listings is the stage cut point calculated from equations C19 and C20 for the actual test conditions. It is labelled $D_{50}$ and is given in micrometer units. The stage weights are likewise listed for the respective stages, labelled MASS and are in milligram units.

The mass loadings per unit volume of gas sampled indicated by the stage weights are labelled MG/DSCM/STAGE and are written in milligrams per dry standard cubic meter. The /STAGE indicates that it is not cumulative. It is calculated for particular stage j by the formula

$$\text{MG/DSCM/STAGE}_j = \frac{\text{MASS}_j}{\text{SAMPLING DURATION (minutes)}}$$

$$\times \frac{\text{35.31 cubic feet/cubic meter}}{\text{FLOWRATE (ACFM)}} \times \frac{\text{Absolute Stack Temperature}}{\text{Absolute Standard Temperature}}$$

$$\times \frac{\text{Absolute Standard Pressure}}{\text{Absolute Stack Pressure}} \times \frac{1}{(1 - \text{Fraction of } H_2O)} \qquad \text{(C26)}$$

where absolute means the temperature and pressure are in absolute units-degrees Rankin or degrees Kelvin for temperature, and atmosphere, inches or millimeters of mercury for pressure. For S1,

$$\text{MG/DSCM/STAGE} = \frac{.72\ \text{mg}}{20\ \text{min}} \times \frac{\text{35.31 cubic feet/cubic meter}}{\text{0.500 ACFM}}$$

$$\times \frac{(400 + 460)^\circ R}{(70 + 460)^\circ R} \times \frac{29.92\ \text{in. Hg}}{26.50\ \text{in. Hg}} \times \frac{1}{(1.0 - 0.01)} = 4.71\ \text{mg/DSCM}$$

The subscripts indicate stage index numbers.

The percent of the mass of particles with diameters smaller than the corresponding $D_{50}$ is called the CUMULATIVE PERCENT OF MASS SMALLER THAN $D_{50}$. It is the cumulative mass at stage j divided by the total mass collected on all the stages, and converted to a percentage:

$$\text{CUM } \%_j = \frac{\sum_{i=j+1}^{9} \text{MASS}_i}{\text{Total Mass}} \times 100 \qquad \text{(C27)}$$

For example, for S6, the cumulative percent is given by

$$\text{CUM }\%_6 = \frac{\text{MASS}_7 + \text{MASS}_8 + \text{MASS}_9}{\text{Total Mass}} \times 100$$

$$= \frac{1.25\text{ mg} + 0.04\text{ mg} + 0.39\text{ mg}}{5.24\text{ mg}} \times 100 = 32.06\%$$

For S8, the mass of the particulate collected on the filter is used,

$$\text{CUM }\%_8 = \frac{\text{MASS}_9}{\text{Total Mass}} \times 100 = \frac{0.39\text{ mg}}{5.24\text{ mg}} \times 100 = 7.44\% \qquad \text{(C28)}$$

Note that the apparent error in the least significant figures of the calculated percentages is due to using masses from the computer printout which have been rounded off to two decimal places before printing.

The cumulative mass loading of particles smaller in diameter than the corresponding $D_{50}$ in milligrams per actual cubic meter (CUM. (MG/ACM) SMALLER THAN $D_{50}$) for a particular stage j is given by the formula

$$\text{CUM. (MG/ACM)}_j = \frac{\sum_{i=j+1}^{9} \text{MASS}_i}{\text{sampling duration (min)}} \times \frac{35.31\text{ cubic feet/cubic meter}}{\text{FLOWRATE (ACFM)}} \qquad \text{(C29)}$$

From the information at the top of the computer printout sheet, the flow rate is 0.500 actual cubic feet per minute (ACFM) and the sampling duration is 20.00 minutes. Therefore, for S4,

$$\text{CUM.(MG/ACM)}_4 = \frac{\text{MASS}_5 + \text{MASS}_6 + \text{MASS}_7 + \text{MASS}_8 + \text{MASS}_9}{\text{20 minutes}}$$

$$\times \frac{\text{35.31 cubic feet/cubic meter}}{\text{0.500 ACFM}} = \text{12.3 mg/ACM}$$

For S8, the mass of the particulate collected on the filter is again used,

$$\text{CUM.(MG/ACM)} = \frac{\text{MASS}_9}{\text{20 minutes}} \times \frac{\text{35.31 cubic feet/cubic meter}}{\text{0.500 ACFM}}$$

$$= \frac{\text{0.39 mg}}{\text{20 minutes}} \times \frac{\text{35.31 cubic feet/cubic meter}}{\text{0.500 ACFM}}$$

$$= \text{1.38 mg/ACM}$$

The cumulative mass loading of particles smaller in diameter than the corresponding $D_{50}$ in grains per actual cubic foot (CUM. (GR/ACF) SMALLER THAN $D_{50}$) for a particular stage j is given by the formula

$$\text{CUM.(GR/ACF)}_j = \frac{\text{CUM.(MG/ACM)}_j}{2.288\ \frac{\text{grams/cubic meter}}{\text{grains/cubic foot}} \times \text{1000 mg/gram}} \qquad \text{(C30)}$$

for S7,

$$\text{CUM.(GR/ACF)} = \frac{\text{1.52 mg/ACM}}{2.288\ \frac{\text{grams/cubic meter}}{\text{grains/cubic foot}} \times \text{1000 mg/gram}}$$

$$= 6.64 \times 10^{-4}\ \text{grains/ACF}$$

The cumulative mass loading of particles smaller in diameter than the corresponding $D_{50}$ in grains per dry standard cubic foot (CUM. (GR/DSCF) SMALLER THAN $D_{50}$) is calculated to show what the above cumulative would be for one cubic foot of dry gas at 70°F and at a pressure of 29.92 inches of mercury. For a particular stage j,

$$\text{CUM.(GR/DSCF)}_j = \text{CUM.(GR/ACF)}_j \times \frac{\text{Absolute Stack Temperature}}{\text{Absolute Standard Temperature}}$$

$$\times \frac{\text{Absolute Standard Pressure}}{\text{Absolute Stack Pressure}} \times \frac{1}{(1\text{-Fraction of } H_2O)} \tag{C31}$$

where absolute means the temperature and pressure are in absolute units-degrees Rankin or degrees Kelvin for temperature, and atmospheres, inches or millimeters of mercury for pressure. For S1,

$$\text{CUM.(GR/DSCF)}_1 = 6.96 \times 10^{-3}\ \text{gr/ACF} \times \frac{(400 + 460)°\text{R}}{(70 + 460)°\text{R}}$$

$$\times \frac{29.92\ \text{in. Hg}}{26.50\ \text{in. Hg}} \times \frac{1}{(1.00-0.01)} = 1.29 \times 10^{-2}\ \text{gr/DSCF}$$

The particle size distribution may be presented on a differential basis which is the slope of the cumulative curve.

Differential size distributions may be derived two ways:

1. Curves may be fitted to the graphs of cumulative mass vs. particle size, and then the differential curves (slope) of each test would be calculated by taking finite differences along the ordinate and abscissa of the fitted curve.

2. The finite differences may be taken equal to the differences in $D_{50}$'s from stage to stage (abscissa) and the particulate mass on each stage (ordinate). This technique was used to calculate the differential size distribution data in Table C3, and is described in detail in the following paragraphs.

If we define the terms:

$\Delta M_j$ = MG/DSCM/STAGE$_j$ and

$(\Delta \log D)_j = \log_{10}(D_{50\,j-1}) - \log_{10}(D_{j})$, then

$$\left(\frac{\Delta M}{\Delta \log D}\right)_j = \frac{\text{MG/DSCM/STAGE}_j}{\log_{10}\left(D_{50\,j-1}\right) - \log_{10}\left(D_{50\,j}\right)} \qquad \text{C32)}$$

Because the computer printer does not contain Greek letters, the computer printout sheet reads DM/DLOGD instead of $\Delta M/\Delta \log D$. For S6,

$$\left(\frac{\Delta M}{\Delta \log D}\right)_6 = \frac{9.35 \text{ mg/DSCM}}{\log_{10}(2.22) - \log_{10}(1.29)} = 39.7 \text{ mg/DSCM}$$

Note that $\Delta M/\Delta \log D$ has the dimensions of the numerator since the denominator is dimensionless. In the calculation for S1, a maximum particle diameter is used. For this example, MAX. PARTICLE DIAMETER = 100.0 micrometers.

$$\left(\frac{\Delta M}{\Delta \log D}\right)_1 = \frac{4.71 \text{ mg/DSCM}}{\log_{10}(100) - \log_{10}(10.74)} = 4.86 \text{ mg/DSCM}$$

For the filter stage, the $D_{50}$ is arbitrarily chosen to be one-half of the $D_{50}$ for stage eight (S8). For this example, it is chosen to be 0.33 micrometers/2 = 0.165 micrometers. Thus,

$$\left(\frac{\Delta M}{\Delta \log D}\right)_9 = \frac{2.55 \text{ mg/DSCM}}{\log_{10}(0.33) - \log_{10}(0.165)} = 8.47 \text{ mg/DSCM}$$

The geometric mean diameter in micrometers GEO. MEAN DIA. (MICROMETERS) for a particular stage j is given by the formula

$$\text{GEO. MEAN DIA.}_j = \sqrt{D_{50\,j} \times D_{50\,j-1}} \qquad \text{(C33)}$$

For S8,

$$\text{GEO. MEAN DIA.}_8 = \sqrt{0.33 \times 0.69} \text{ micrometers}$$
$$= 0.477 \text{ micrometers}$$

As in the ΔLOGD calculation, we again use the maximum particle diameter for the stage one calculation and one-half the $D_{50}$ for stage eight for the filter stage calculation.

For S1,

$$\text{GEO. MEAN DIA.}_1 = \sqrt{10.74 \times 100.0} \text{ micrometers}$$
$$= 32.8 \text{ micrometers}$$

For the filter,

$$\text{GEO. MEAN DIA.}_9 = \sqrt{0.165 \times 0.33} \text{ micrometers}$$
$$= 0.23 \text{ micrometers}$$

A differential number distribution (for comparison with ultrafine data) can also be derived. Since $\Delta M_j$ = MG/DSCM/STAGE$_j$ is the mass per unit volume for stage j, then we can define $\Delta N_j$ as $\Delta N_j$ = NUMBER OF PARTICLES/DSCM/STAGE$_j$ or the number of particles per unit volume for stage j. Now $\Delta M_j$ and $\Delta N_j$ are related by the equation $M_J = N_j \times m_p$, where $m_p$ is the average mass of the particles collected on one stage. Dividing both sides of the equation by $m_p \times \Delta\log D$ yields

$$\frac{(\Delta M/\Delta \log D)_j}{m_p} = \left(\frac{\Delta N}{\Delta \log D}\right)_j \tag{C34}$$

Now $m_p = \rho_p V_p$ where $\rho_p$ is the assumed particle density and $V_p$ is the average volume of one particle on one stage. To obtain $m_p$ in milligram units when $\rho_p$ is in grams per cubic centimeter and $V_p$ is in cubic micrometers, certain conversion factors must be used. The complete formula, using the correct conversion factors and the expression $(4/3)\ (\pi)\ (d/2)^3$ for $V_p$ where d is the geometric mean diameter in micrometers, is:

$$m_p = \rho_p \left(\frac{10^3\ mg}{1\ gm}\right)\left(\frac{4\pi}{3}\right)\left(\frac{d}{2}\right)^3\left(\frac{10^{-12}\ cm^3}{1\ \text{cubic micrometer}}\right)$$
$$= 5.23599 \times 10^{-10}\ \rho_p d^3.$$

Therefore,

$$\left(\frac{\Delta N}{\Delta \log D}\right)_j = \frac{(\Delta M/\Delta \log D)_j}{5.23599 \times 10^{-10} \rho_p d^3}$$

where $\Delta M/\Delta \log D$ is in units of mg/DSCM, $\rho_p$ is in gm/cm³, d is in microns, and $\Delta N/\Delta \log D$ is in number of particles/DSCM. For S3,

$$\left(\frac{\Delta N}{\Delta \log D}\right)_3 = \frac{17.9\ mg/DSCM}{(5.23599 \times 10^{-10}) \times (1.35\ gm/cc) \times (7.96\ microns)^3}$$
$$= 5.02 \times 10^7\ \text{particles/DSCM}.$$

For the filter stage,

$$\left(\frac{\Delta N}{\Delta \log D}\right)_9 = \frac{8.47\ mg/DSCM}{(5.23599 \times 10^{-10}) \times (1.35\ gm/cc) \times (0.231\ microns)^3}$$
$$= 9.72 \times 10^{11}\ \text{particles/DSCM}$$

The test data are usually classified according to sampling location (outlet or inlet), sampling time (day, week, etc.) and combustion chamber or pollution control device conditions (high or low sulfur coal for coal plants, normal or below normal fuel consumption, etc.). When classified, all of the data taken in a single classification are usually averaged and plotted on appropriate graph paper.

Curves are drawn through the discrete points. At selected particle sizes values of ΔM/ΔlogD and ΔN/ΔlogD are chosen from these curves for averaging.

Figure C5 shows a typical average ΔM/ΔlogD outlet particle size distribution curve, from Andersen impactor data, and Figure C6 shows the same data presented as ΔN/ΔlogD.

Figure C7 shows similar data for a hypothetical inlet test when a Brink impactor was used. The size distributions of Figures C5 or C7 were used to calculate the penetration/efficiency curve of Figure C8. Confidence limits were calculated for the average data as described in Section C.8.

A computer program (Johnson, 1978) has been written which does all of the calculations necessary to generate plotted graphs of the particle size distributions and fractional efficiency curves from the raw field data. Table C4 shows a flow diagram for this program. Copies of the report describing this computer program are available from the EPA or NTIS.

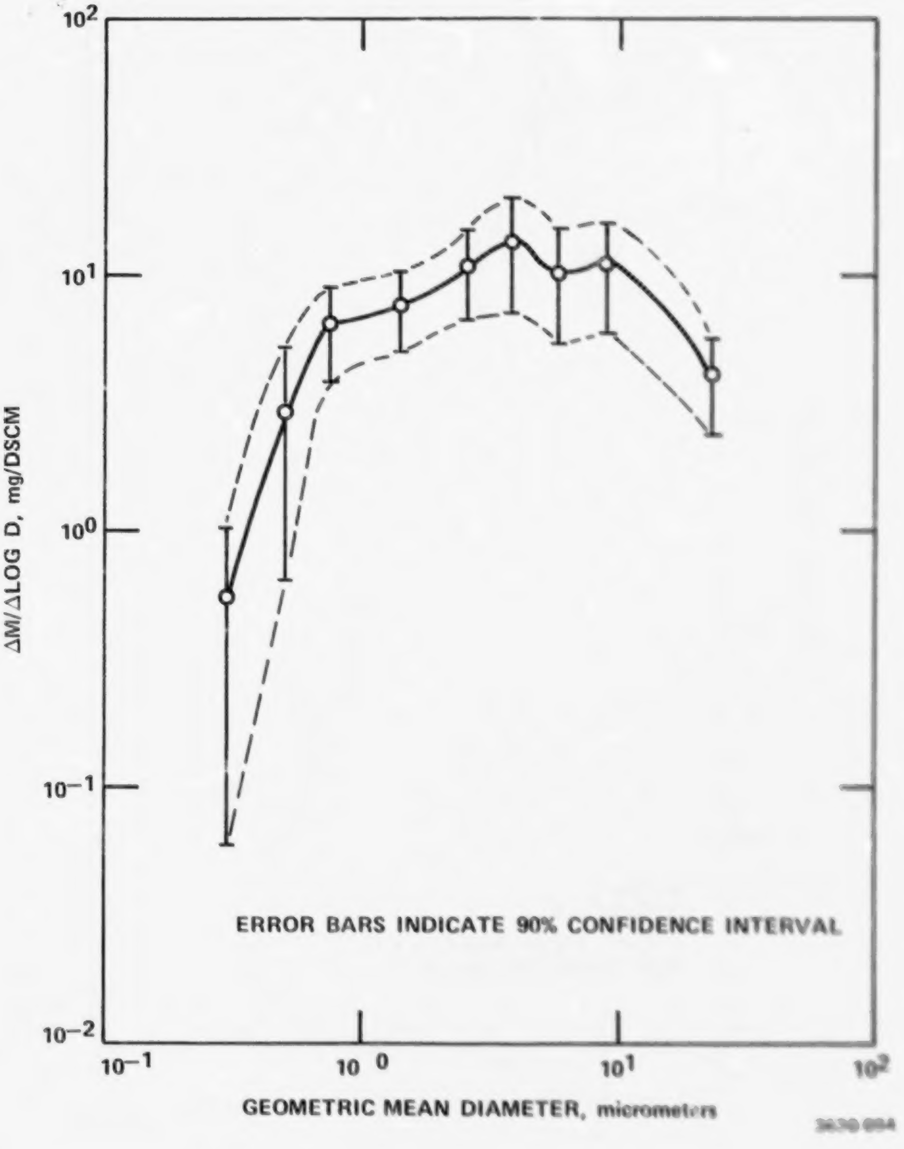


*Figure C5. Hypothetical particle size distribution at a fabric filter outlet determined from Andersen impactor data.*

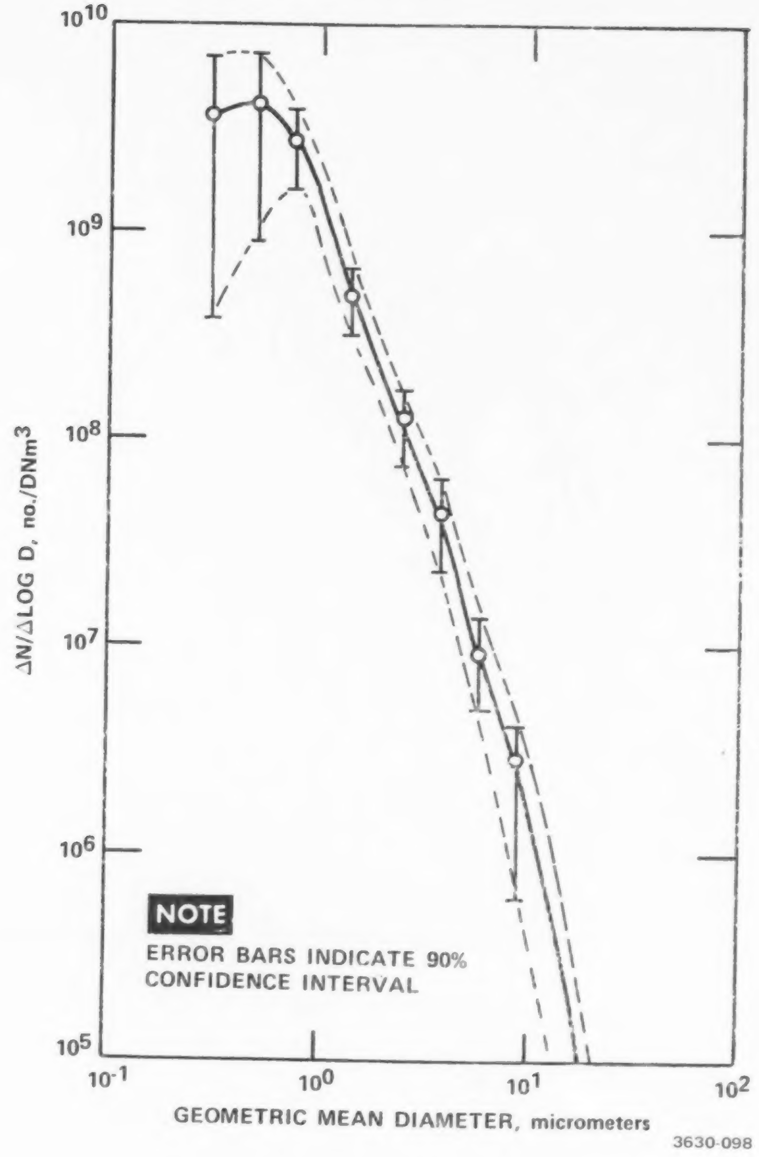


*Figure C6. Hypothetical particle size distribution at a fabric filter outlet determined from Andersen impactor data.*

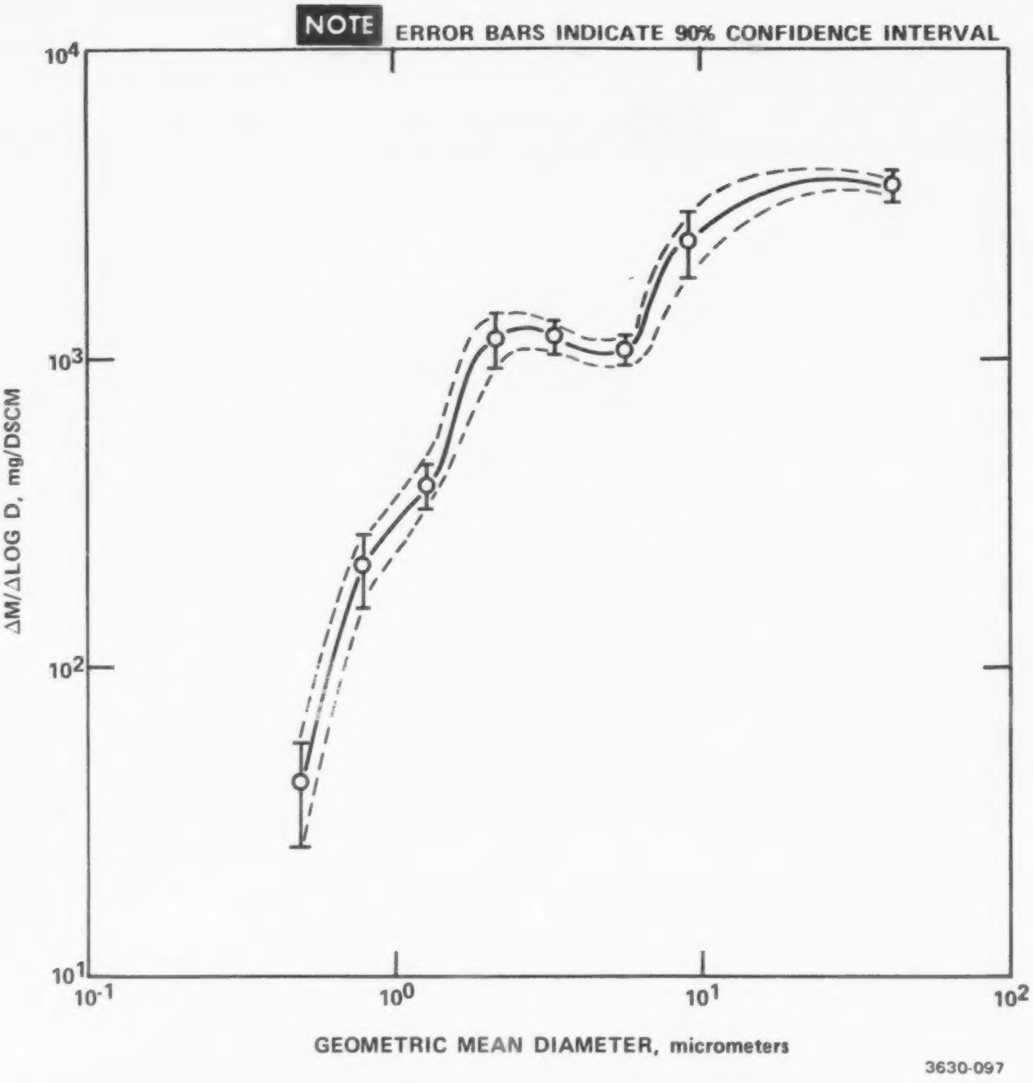


*Figure C7. Hypothetical particle size distribution at a fabric filter inlet determined from Brink impactor data.*

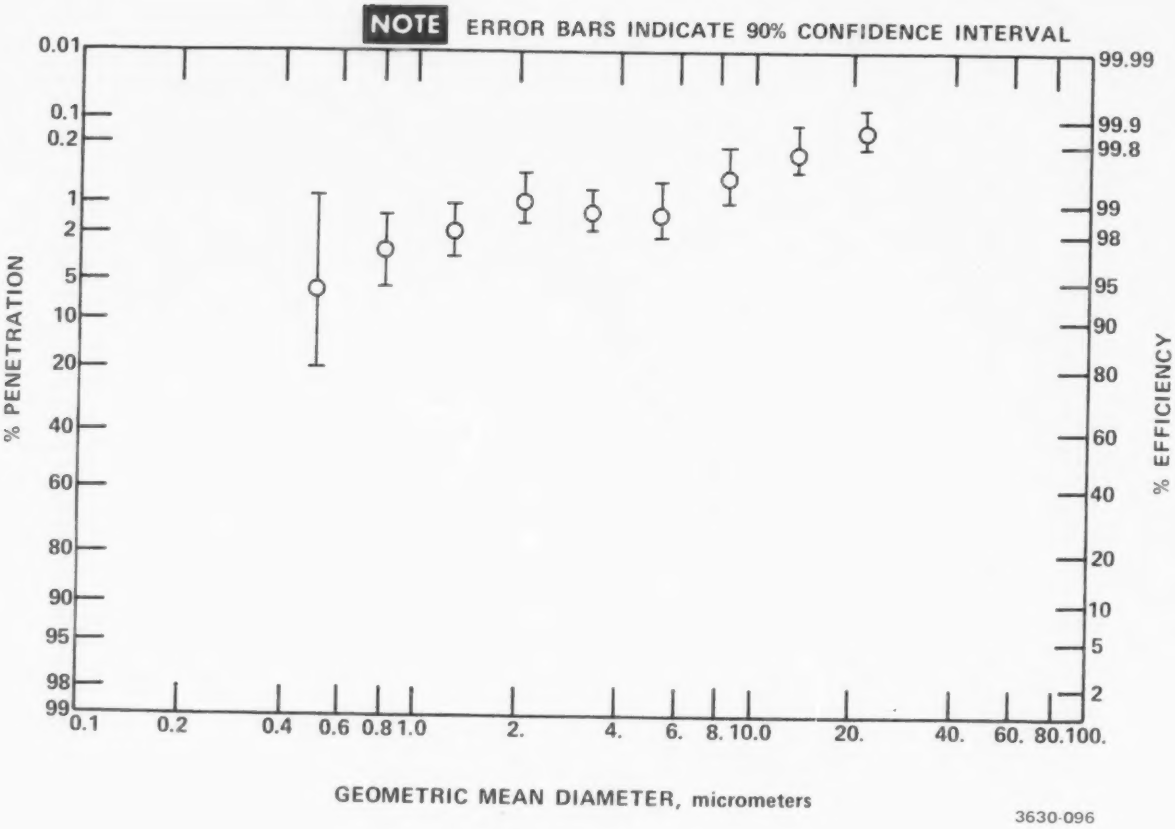


*Figure C8. Hypothetical fabric filter fractional efficiency based on the data presented in Figures C5 and C7.*

# TABLE C4

# PROGRAM FLOW

For all inlet data:

## I. Impactor Program (MPPROG)

Takes testing conditions, stage weights, and impactor constants to produce stage $D_{50}$'s, cumulative and cumulative % mass concentrations $< D_{50}$, geometric mean diameters, and mass and number size distributions.

## II. Fitting Program (SPLIN1)

Uses modified spline technique to fit cumulative mass loading points for each plot. Stores fitting coefficients and boundary points on file.

## III. Graphing Program (GRAPH)

Produces individual run graphs with points based on stage weights and impactor $D_{50}$'s. Also superimposes plot based on fitted data, if desired. Graphs include cum. mass loading, cum. % mass loading, and mass and number size distributions.

## IV. Statistical Program (STATIS)

Recalls cum. mass loading fitting coefficients to produce avg. cum. mass loading, avg. % cum. mass loading, avg. mass size distribution, and avg. number size distribution plots each with 90% confidence bars.

Repeat programs I-IV for outlet data.

## V. Efficiency Program (PENTRA)

Recalls avg. mass size distribution values along with 90% confidence limits for inlet and outlet to plot percent penetration and efficiency with 90% confidence bars.

# APPENDIX D
# SIZE DISTRIBUTIONS OF SUBMICRON AEROSOL PARTICLES

If it is desirable to measure the fabric filter collection efficiency for ultrafine particles, measurements of the particle size distribution must be made at the inlet and outlet. This appendix describes procedures for making measurements of the particle size and concentration from 0.01 to 2 μm diameter.

## D.1 SYSTEM FOR EXTRACTIVE SAMPLING

When possible, in-stack sampling is preferred because it eliminates many condensation and sample loss problems which occur when probes are used for extractive sampling. Unfortunately, existing submicron sizing techniques and instrumentation are designed for a laboratory environment and cannot be used in-stack.

Particulate concentrations are usually extremely high in industrial flues and vary by orders of magnitude from one industrial process to another and from the inlet side of a control device to the outlet of the same device. Temperature, pressure, moisture content, and the physical properties of the particulate also vary widely from one industrial process to another. Because of this complexity and the limited useful concentration range for particle sizing techniques, extensive sample dilution and conditioning is required to obtain information on submicron particles in an industrial gas stream.

### D.1.1 Line Losses

When extracting the sample, attention should be given to line losses which can be a problem for particles smaller than 0.01 μm or greater than 1.5-2.0 μm diameter. Since the objective is to measure the concentration of ultrafine particles, there is little interest in measuring particles larger than about 2.0 μm. Thus, losses due to impaction and settling are not significant, and isokinetic sampling is unnecessary. Diffusional and electrostatic line losses are of concern, however. For example, at a sampling rate of 1 ℓpm, a sample line will remove 0.005 μm particles by diffusion at a rate of about 8% of the instantaneous concentration for every foot of sample line, independent of the radius of the sample line. This problem can never be eliminated for the case of out-of-stack sampling, but it can be minimized by (1) using short probes and as high a flow rate as is practical, or (2) by using high, nonturbulent, flow rates through a probe and connecting lines to a conditioning-dilution system in which a sample is split off at the required rate. This second technique allows one to use long probes and change sampling points without disconnecting and reconnecting all sampling lines. Diffusional line losses for non-turbulent flow can be estimated from the equations for diffusion to the walls in a circular geometry (Figure D1). A similar discussion of diffusional line losses has been given by Ensor and Jackson (1975).

### D.1.2 Condensation of Gases

Another problem of concern is condensation. Elements which are at a gaseous state at stack temperature ($SO_3/H_2SO_4$ in particular) can drop below their dew point and form high concentrations of very small particles resulting in anomalously high readings. In the case of $SO_3$ in the presence of $H_2O$, a sulfuric acid fume can be formed if temperatures fall below the acid dew point

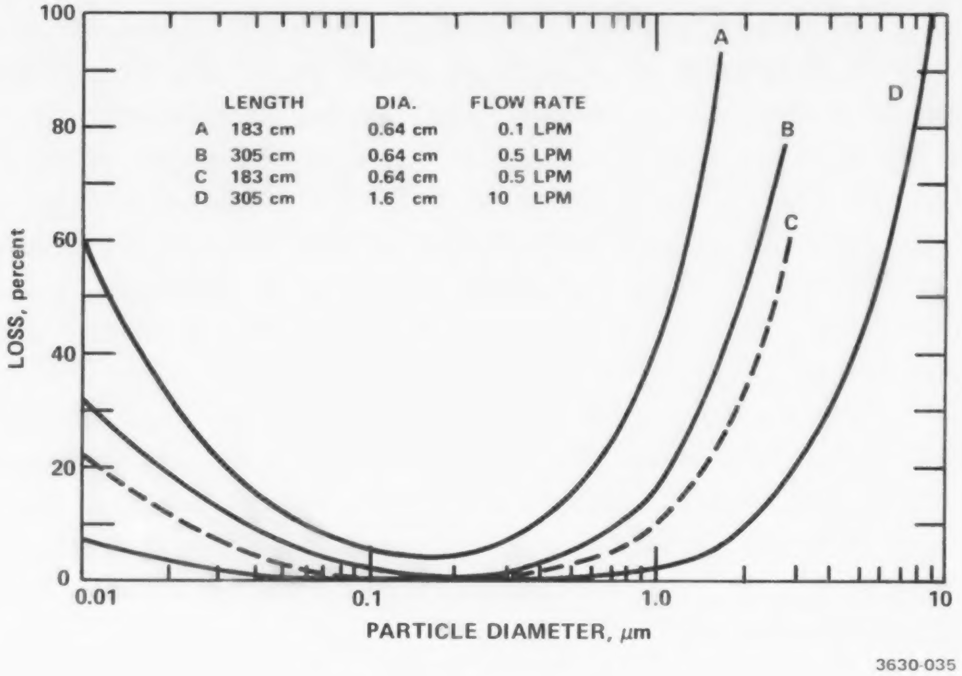


*Figure D1. Probe losses due to settling and diffusion for spherical particles having a density of 2.5 gm/cm$^3$ under conditions of laminar flow.*

(temperature, pressure, and concentration sensitive). Once this fume has been formed, very high temperatures are required to re-evaporate the droplets. For this reason temperatures above the dew point must be maintained throughout the system until the gaseous $SO_3/H_2SO_4$ can be removed or diluted. Two techniques appear to be useful for doing this: (1) diffusion to an absorber reagent and (2) dilution of the $SO_3/H_2SO_4$ while hot, to levels at which the mist will not be formed (Sem, 1975). The main problem with the second technique, however, is that a further dilution is required to bring the aerosol to a temperature within the operating range of the instrument. As a result, the minimum total dilution, which is the product of hot stage and cold stage dilutions, may be excessive for low particulate concentration levels such as those found at the outlet of some gas cleaning devices. Hence the concentration of ultrafine particles even at the minimum dilution may be below the minimum detection level of the sizing instrument.

At low levels of $SO_3$, such as those at power plants burning low sulfur coal, copper has been found to be a successful $SO_3/H_2SO_4$ absorber reagent through the formation of $CuSO_4$. This reagent has the particular advantage that the reaction product is water soluble, and the absorber can easily be rejuvenated. Activated charcoal is also a very effective absorber of $SO_x$, even at relative high concentrations.

A special effort must be made to detect any particles generating interferences and to eliminate these if possible by conditioning the extracted sample. Condensation may be observed by periodically checking the linearity of the dilution system. When the dilution system is adjusted to produce a many-fold change in dilution, the indicated concentration should reflect an equal change in measured concentration.

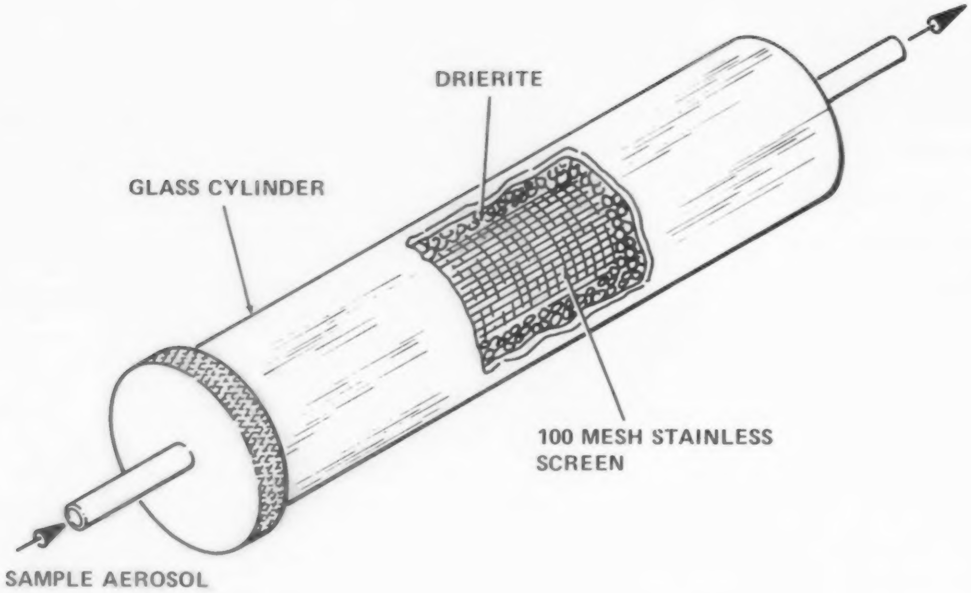
Figure D2 shows a diffusional absorber/dryer for the removal of water vapor from the sample stream. Figure D3 shows a sample extraction system which includes diffusional absorbers for high temperature use.

### D.1.3 Temperatures

Consideration must also be given to reducing the gas temperature to a level at which the instruments were designed to operate. This is normally done by using a large volume of cool, dry dilution air but in some cases can be done by simply pulling the sample through an ice bath condenser. It should be remembered, however, that unless condensible gases have previously been removed, it is possible to form a condensation fume when using a simple condenser.

### D.1.4 Electrostatic Losses

Electrostatic line losses can also be a problem. Due to charges present on the particles, large static electric fields can be established which result in particle deposition and nonrepresentative sampling. Line losses may be more severe if the aerosol particles are charged. Electric fields may exist which result in particle deposition and nonrepresentative sampling. This problem is most severe if the sample lines are made of insulating materials. Particle losses may also be a problem in the diffusion batteries where the theory assumes that the particles are electrically neutral and no consideration is given to unknown electrical forces. Also, it is assumed in the application of the electrical aerosol analyzer that the sample aerosol particles initially bear no charge. Precharged particles could acquire charges different from those of calibration, causing them to exhibit different mobility vs size characteristics. It is desirable to neutralize the particle charge prior to entering


DRIERITE
GLASS CYLINDER
100 MESH STAINLESS SCREEN
SAMPLE AEROSOL

3630-05

*Figure D2. Diffusional adsorption apparatus for removal of* $H_2O$ *from sample aerosol.*

the probe nozzle, and charge neutralization to Boltzmann equilibrium can be accomplished by exposure to an ion field created by radioactive materials, however, no suitable radioactive source has been developed for in-stack applications. The approach generally taken has been to use radioactive materials such as $P_0^{210}$ in the diluter to neutralize the particulate after it has been cooled and before it goes to the sizing instruments. The extent of electrostatic interferences, however, has not been well quantified.

### D.1.5 Humidity

High humidities can alter the charging characteristics of electrical mobility analyzers and can cause water to condense on particles and change their size (similar to the controlled process occurring in a condensation nuclei counter). Humidity problems can be eliminated by the use of (1) dry dilution air or (2) diffusional dryers.

### D.1.6 Dilution

In-stack concentrations generally exceed instrument concentration limits and must be reduced to levels at which the instrument functions properly. Large changes in the aerosol size distribution due to coagulation are also of concern, particularly in a diffusional configuration involving long residence times, since the loss rate due to coagulation for a given size particle rises rapidly with increasing concentration.

Most of the dilution systems used to date involve the measurement of two different flow rates. Several methods are available for doing this: (1) Rotameters, (2) Orifices, (3) Venturis, and (4) Mass Flowmeters. Spink (1957) has summarized the use of the first three and Parry and Meyer (1974) have described the use of several mass flowmeters in an automated system. Rotameters are convenient for measuring cool dilution air flow rates but could cause sample losses when measuring sample gas flows. They are also sensitive to temperature, pressure, and gas composition. Flowmetering orifices are useful for measuring flow rates of hot gases but significant sample losses can occur for particles larger than about 2 μm. Orifices also require, as do venturis, that pressure drops, etc. be monitored in order to calculate the flow rate. Venturis have the advantage that less turbulence occurs in the meter and hence size dependent losses should be much less than those for orifices. Venturis have the disadvantage that the pressure tap must be taken at the Vena Contracta making them difficult to construct for low flow rates. Mass flowmeters are ideal but may require line restrictions in order to throttle the flow rate.

### D.1.7 Sample Extraction-Dilution Systems (SEDS)

Figure D3 is a block diagram of a sample extraction-dilution system (SEDS) developed by Southern Research Institute under EPA Contract No. 68-02-2114. In this system a 233 $cm^3$/sec sample flow is removed from the process exhaust stream and is pulled through a rigid probe, a flexible connector hose, and a cyclone, into a "T" where the flow splits. The excess flow is dumped and the desired sample flow goes into the diluter via a calibrated orifice and an optional bank of sulfur oxide absorbers. The cyclone, orifice, and sulfur oxide absorber bank are housed in a heated box so that all components of the system except the diluter can be maintained at the stack temperature (up to 200°C) to prevent condensation. Pressure taps for the cyclone and the orifice allow continuous monitoring of the cyclone flow rate and the orifice flow rate by reading the pressure drop across the respective component.

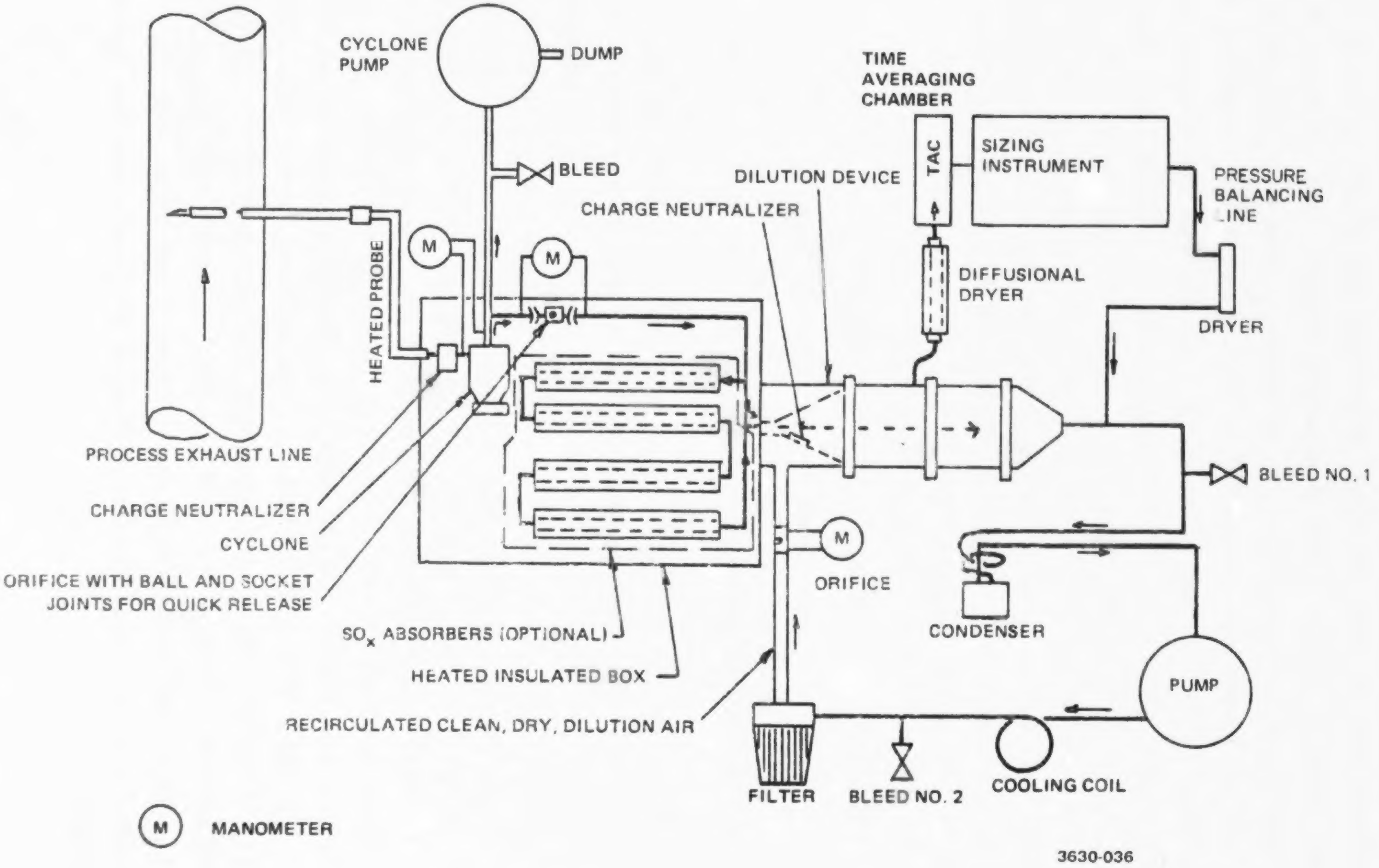

CYCLONE PUMP
DUMP
BLEED
TIME AVERAGING CHAMBER
TAC
SIZING INSTRUMENT
PRESSURE BALANCING LINE
DILUTION DEVICE
CHARGE NEUTRALIZER
DIFFUSIONAL DRYER
DRYER
M
M
HEATED PROBE
PROCESS EXHAUST LINE
CHARGE NEUTRALIZER
CYCLONE
ORIFICE WITH BALL AND SOCKET JOINTS FOR QUICK RELEASE
BLEED NO. 1
M
ORIFICE
CONDENSER
$SO_x$ ABSORBERS (OPTIONAL)
HEATED INSULATED BOX
PUMP
RECIRCULATED CLEAN, DRY, DILUTION AIR
FILTER
BLEED NO. 2
COOLING COIL
M
MANOMETER
3630-036

Figure D3. Sample Extraction - Dilution System (SEDS)

Currently, charge neutralization is done in the cone of the diluter by two 500 μC Polonium-210 strips, mounted as shown. The sample gas enters the dilution chamber at the apex of a perforated cone into which clean, dry air is pumped through the perforations, creating a highly turbulent mixing zone. Calibration data for the diluter, Figure D4, is shown in Figures D5 and D6. At a downstream point, after adequate mixing has occurred, the diluted sample is extracted and conveyed to the sizing instrument. This diluted sample passes through a diffusional dryer where any remaining moisture is removed. The major drying action is accomplished by using dilution air which has been passed through an ice bath condenser.

Just prior to entering the sizing instrument, the sample passes through a plenum to damp out short term concentration changes. The exhaust from each instrument is returned to the diluter to reduce pressure drops across the sizing devices. If further drying of this recycled gas is necessary, absorption driers are placed in the instrument exhaust lines.

Changing the sample air flow and the dilution air flow allows one to change the dilution ratio. Sample air and dilution air flowmeters are controlled by two bleed valves on the dilution air pump, one upstream of the pump (#1) and one downstream (#2). Manipulation of these valves changes the internal pressure of the diluter which, in turn, sets the sampling rate. As the pressure in the diluter is reduced, the sample flow rate is increased. In practice, the operation of these valves changes the dilution air flow only about 10% for a many-fold change in sample flow.

McCain, et al (1974), has reported some problems with an earlier prototype conditioning system; growth of particles when high concentrations of $SO_3$ are present, pluggage of orifices, single point sampling limitations, and diffusional losses in

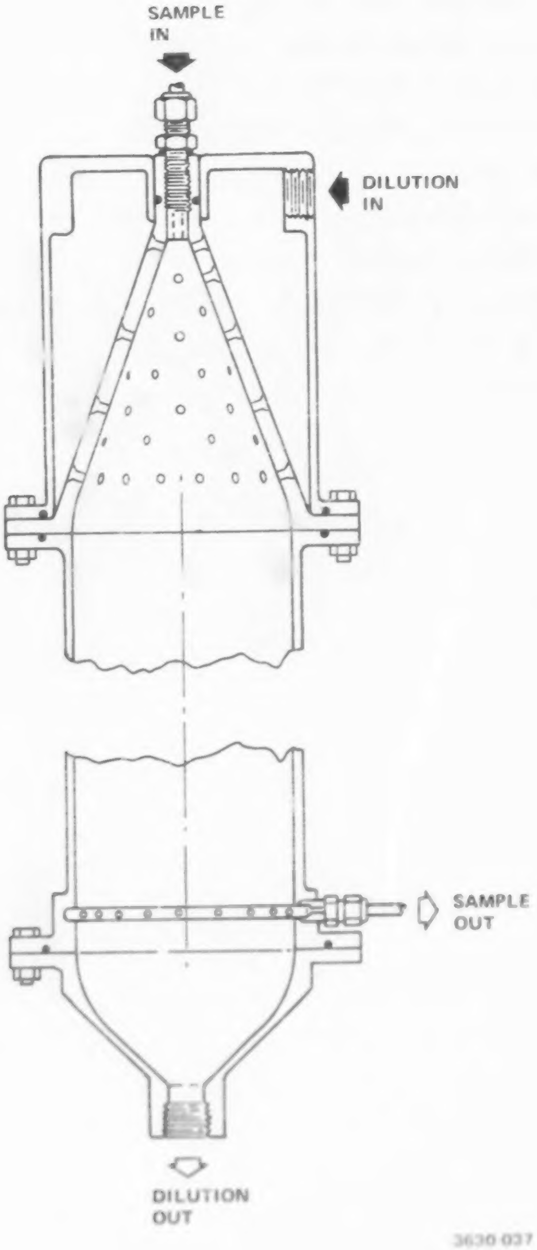

SAMPLE IN
DILUTION IN
SAMPLE OUT
DILUTION OUT
3630-037

*Figure D4. Sample Extraction Diluter, cut-away view.*

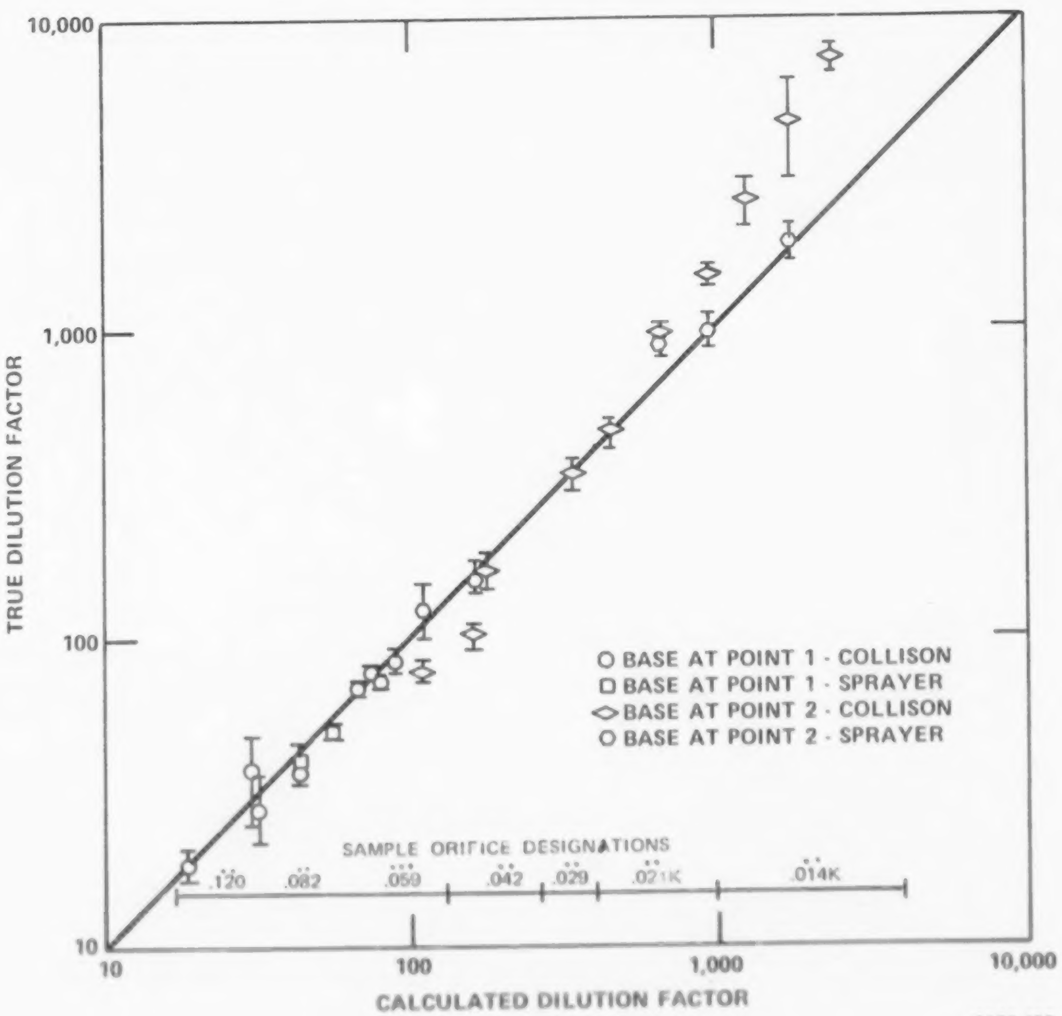


*Figure D5. Calculated dilution versus true dilution for the Southern Research Institute Ultrafine Particle Diluter, 0.092 μm particles.*

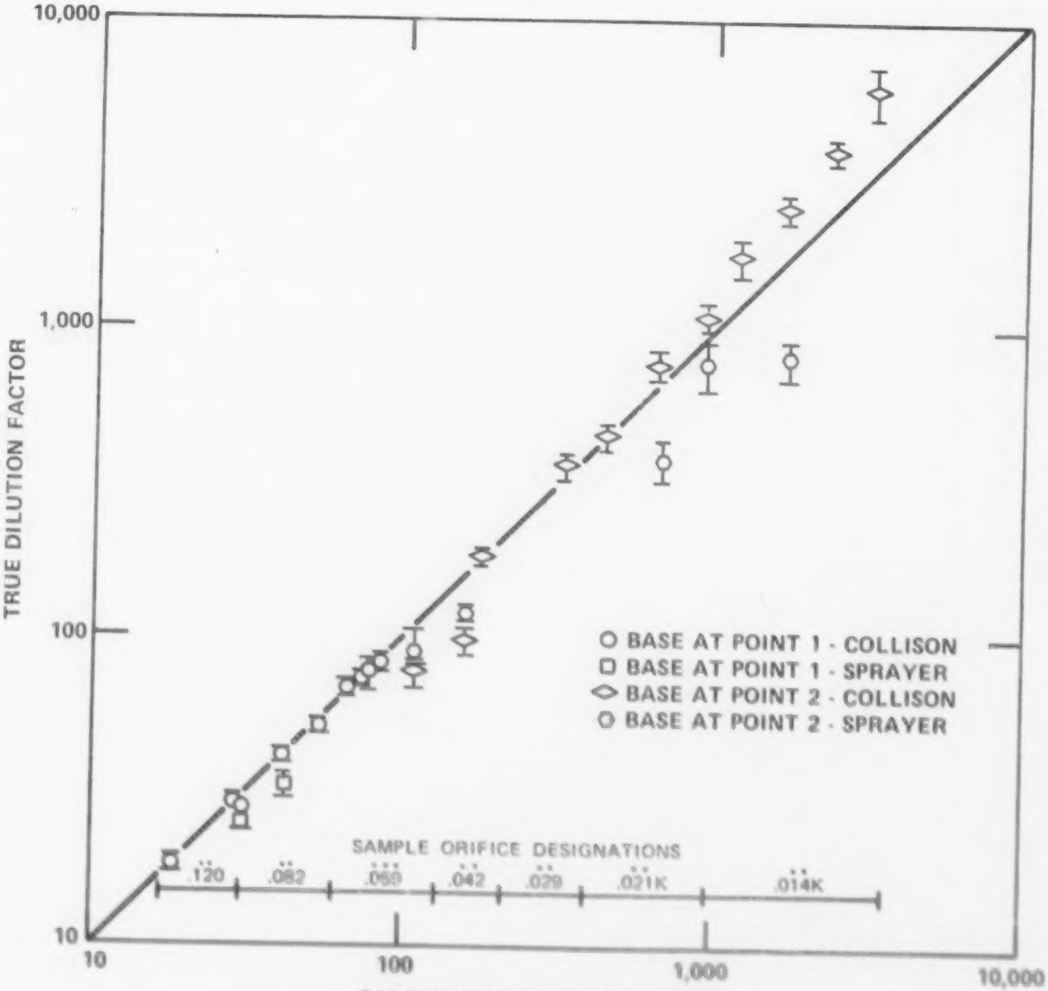


Figure D6. Calculated dilution versus true dilution for the Southern Research Institute Ultrafine Particle Diluter, 0.15 μm particles.

sampling lines. The configuration described above allows the use of optional $SO_x$ absorber chambers, rapid replacement for plugged orifices, quick positive determination of partial pluggage, full traverse sampling capability, and decreased sample line losses. The hot box configuration also increases the total time available for data acquisition by decreasing the time needed to change orifices or dilution ratios (2 minutes compared to about 30 minutes).

Different sampling and dilution systems have been developed and reported by Ensor and Jackson (1975), Bradway and Cass (1975), and Schmidt _et al_ (1976).

It is clear that the most difficult problem in making size distribution measurements of submicron particles is extracting a representative sample from the duct and conditioning it for compatibility with the sizing instruments.

A variety of instruments are available to characterize the conditioned sample, and these are described in the remainder of this appendix.

## D.2 SUBMICRON PARTICLE SIZING TECHNIQUES BASED ON PARTICLE DIFFUSIVITY

### D.2.1 Diffusion Batteries

Fuchs (1954) has reviewed diffusional sizing work up until 1956, while Sinclair (1972, 1975), Sinclair and Countess (1975), Breslin _et al_ (1971), Twomey (1963), Thomas (1955), and Sansone and Weyel (1971) have reported more recent work, both theoretical and experimental.

Diffusion batteries may consist of a number of long, narrow, parallel channels, a cluster of small bore tubes, or a series of screens. Variations in the length and number of channels (tubes, or screens) and in the aerosol flow rate are used as a means of measuring the number of particles in a selected size range. As the aerosol moves in streamline flow through the channels, the particles diffuse to the walls at a predictable rate depending on the particle size and the diffusion battery geometry. It is assumed that every particle which reaches the battery wall will adhere; therefore, only a fraction of the influent particles will appear as the effluent of a battery. It is only necessary to measure the total number concentration of particles at the inlet and outlet to the diffusion battery under a number of conditions in order to calculate the particle size distribution.

When the Stokes diameter is used to describe particle size, the penetration of diffusion batteries is virtually independent of physical properties of the individual aerosol particles.

Parallel plate geometry -- The parallel plate geometry is convenient because of ease of fabrication and the availability of suitable materials, and also because sedimentation can be ignored if the slots are vertical, while additional information can be gained through settling if the slots are horizontal. See Figure D7. Disadvantages of the parallel plate diffusion batteries are (1) the bulk of the diffusional batteries, and (2) the long transport time required to measure a size distribution.

The mathematical expression for the penetration of a rectangular slot or parallel plate diffusion battery by a monodisperse aerosol was given in series form by Twomey (1963).

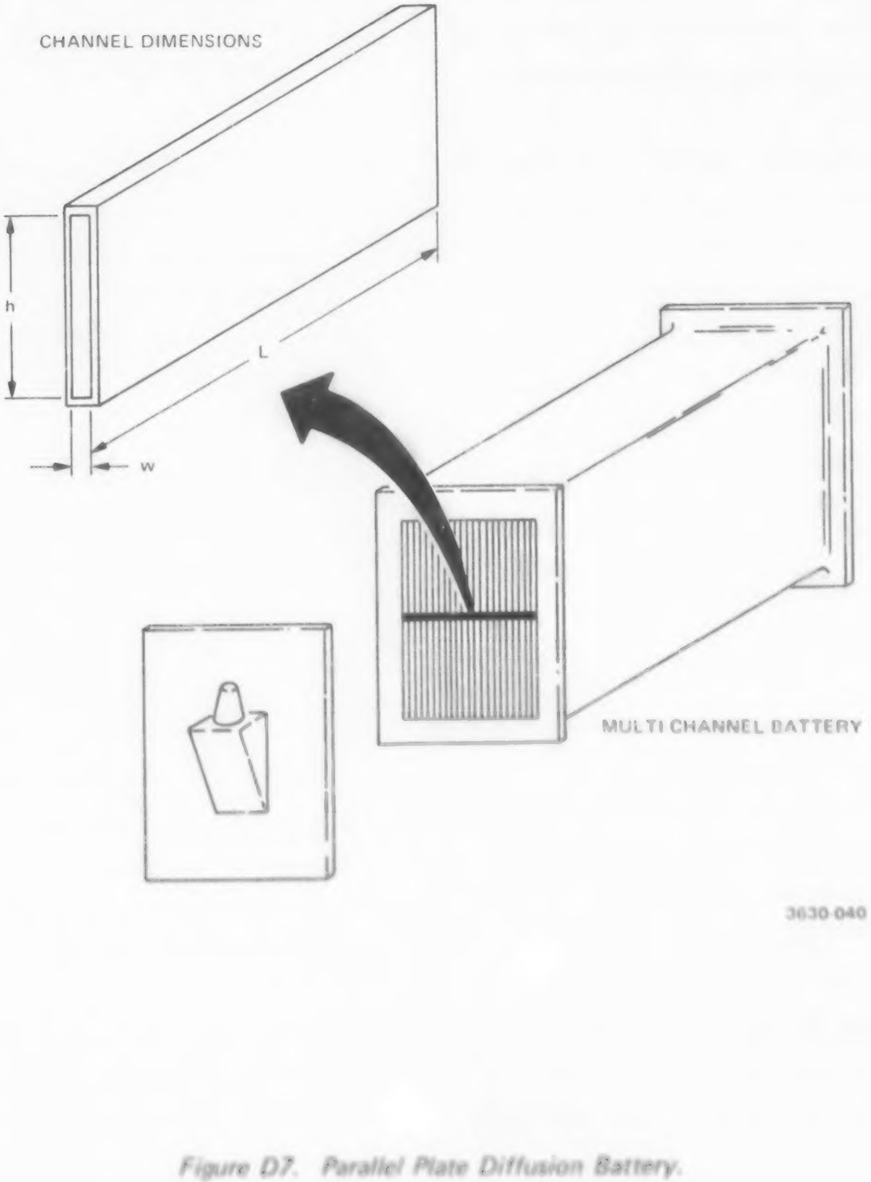

CHANNEL DIMENSIONS
h
L
w
MULTI CHANNEL BATTERY
3630-040

Figure D7. Parallel Plate Diffusion Battery.

By varying the number of diffusion batteries in series and the flow rate, it is possible to measure penetrations under a variety of conditions. Using a set of diffusion batteries such as those developed at Southern Research (four-98 channel diffusion batteries, and a 13 channel diffusion battery) and measuring the penetration at three different flow rates, yields fifteen data points from which the particle size distribution (0.01-0.2 μm diameter) can be reconstructed.

When calculating the fraction ($n/n_0$) of the aerosol which penetrates a series of diffusion batteries, the transport time through the diffusion batteries must be taken into account. This transport time is about 3½-5 minutes for a 98 channel, parallel plate diffusion battery. Thus, with four of these diffusion batteries in series, n, at a particular time, would be related to $n_0$ at a point 16-20 minutes earlier in time on a chart recording. Making a complete measurement of a particle size distribution requires 2-4 hours and diffusional measurements are the most useful on stable sources where the distribution is constant in time when using parallel plate diffusion batteries. The transport time is determined by the open air volume of the diffusion battery and flow rate. If the same geometry constant can be obtained with smaller open air volumes, the transport time can be reduced. This can be accomplished by decreasing the plate spacing, but one very quickly approaches problems with material flatness errors, or equipment survivability when such materials as precision ground graphite sheets are used. These problems seem to have been eliminated with the screen geometry, which is discussed below.

Screen geometry -- Breslin et al (1971) and Sinclair (1972) report success with more compact, tube-type and screen-type arrangements in laboratory studies.

Although the screen-type diffusion battery must be calibrated empirically, it offers convenience in cleaning and operation, and compact size. Figure D8 shows Sinclair's geometry. This battery is 21 cm long, approximately 4 cm in diameter, and weighs 0.9 kg.

This system is commercially available from TSI Incorporated, St. Paul, MN 55113 as the Model 3040 Diffusion Battery. Because of the small internal volume of the battery, the time necessary to obtain a test on one battery is reduced by about a factor of ten as compared to the parallel plate batteries. This diffusion battery system allows data to be collected on process streams where the particle concentration is somewhat unstable (∼15 minutes per cycle).

A disadvantage of the small volume is the sensitivity to surges in flow rate caused by the commercially available CNC counter.

### D.2.2 Particle Concentration Indicators-Condensation Nuclei Counters (CNC)

Condensation nuclei counters function on the principle that particles act as nuclei for the condensation of water or other condensable vapors in a supersaturated environment. This process is used to detect and count particles in the 0.002 to 0.3 micron range (often referred to as condensation or Aitken nuclei). In condensation nuclei detectors, a sample is withdrawn from the gas stream, humidified, and brought to a supersaturated condition by reducing the pressure. In this supersaturated condition, condensation will be initiated on all particles larger than a certain critical size and will continue as long as the sample is supersaturated. This condensation process forms a homogeneous aerosol, predominantly composed of the condensed vapor containing one drop

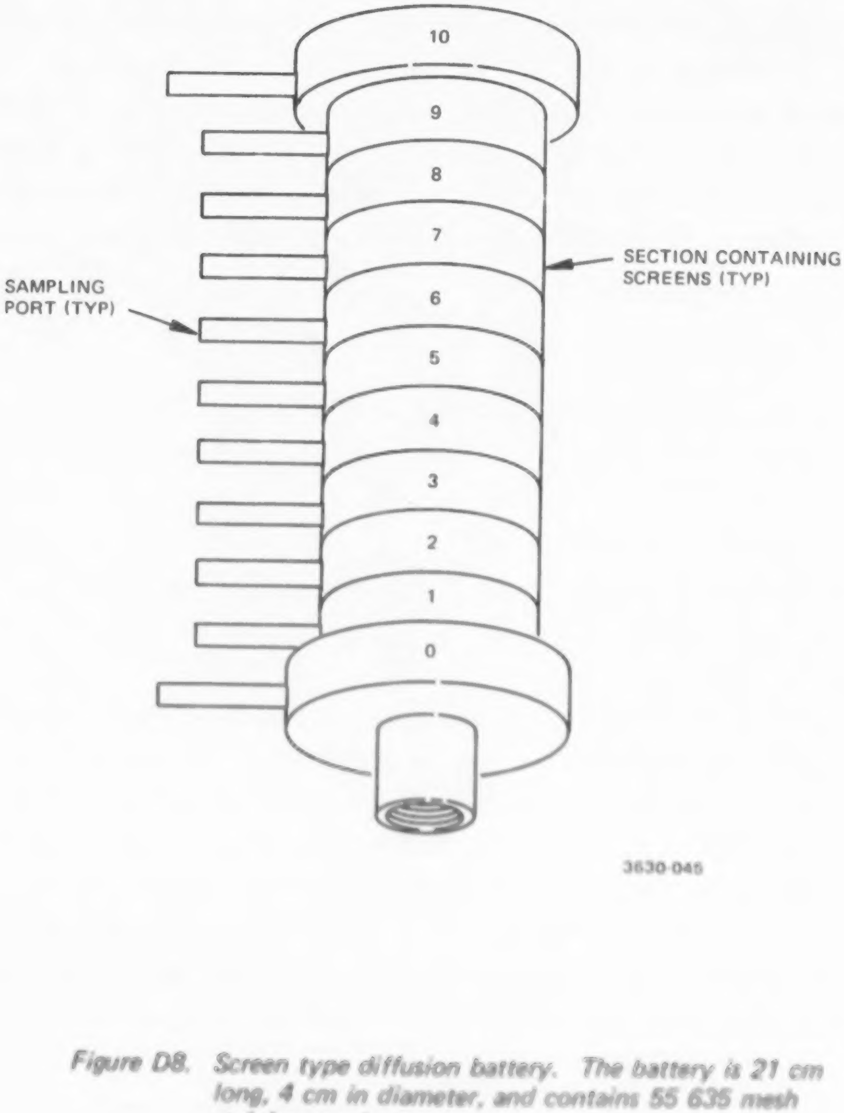

10
9
8
7
6
5
4
3
2
1
0
SAMPLING PORT (TYP)
SECTION CONTAINING SCREENS (TYP)
3630-045

*Figure D8.* *Screen type diffusion battery. The battery is 21 cm long, 4 cm in diameter, and contains 55 635 mesh stainless steel screens.*

for each original particle whose size was greater than the critical size appropriate to the degree of supersaturation obtained; a greater degree of supersaturation is used to initiate growth on smaller particles. The number of particles that are formed is estimated from the light scattering properties of the final aerosol.

Because of the nature of this process, measurements of very high concentrations can be in error as a result of a lack of correspondence between particle concentration and scattering or attenuation of light. Additional errors can result from depletion of the vapor available for condensation. Certain condensation nuclei measuring techniques can also obtain information on the size distribution of the nuclei; that is, variations in the degree of supersaturation will provide size discrimination by changing the critical size for which condensation will occur. However, MacLauren and Junge (1971) have predicted that the critical size for initiating condensation is also affected by the volume fraction of water soluble material contained in the original aerosol particle, so the critical size will be uncertain unless the solubility of the aerosol particles is known. At very high degrees of supersaturation (about 400%), solubility effects are only minor and essentially all particles in the original aerosol with diameters larger than 0.002 μm will initiate the condensation process.

A continuous flow CN counter has been described by Sinclair and Hoopes (1975) and an absolute calibration of a CN counter has been done by Liu and Pui (1974). The theory and principles of operation of CN counters has been described by Haberl (1970) and Haberl and Fusco (1970).

### D.2.3 Using Diffusion Batteries With Condensation Nuclei Counters and a Sample Extraction-Dilution System To Measure Concentrations of Submicron Aerosols In Industrial Flue Gases

Before taking the equipment into the field, a preliminary examination of the sampling site should be made. There must be ample space for the diffusion batteries (D.B.) and the condensation nuclei counter (CNC). The sample lines to and from the D.B.'s should be as short as is practical. The diffusion batteries should be placed out of direct sunlight to reduce thermal interferences.

Four models of CNC's are presently used. Two automatic models are the General Electric (GE)* and the Environment One Model Rich 100 (E-1).** Small manual particle detectors are also commercially available from Gardner Associates*** and Environment One.** The valving system in the GE is mechanical, and pressure differentials across the valves are permissible. However, the E-1 has pneumatic valves and a pressure difference of greater than 2 inches of water across the valves will lock them either open or shut. Soderholm (1976) has described some modifications to the E-1 Rich 100 that replace the pneumatic valves with solenoid valves and smooth out pulsations in the sample flow. A return line is normally used on the GE but may not be connected to the diluter in some circumstances. The GE creates substantial pulsation in the sample lines which is intolerable for diffusional analysis, and an antipulsation device consisting of two metal cylinders connected by a small orifice may be used as a pneumatic R-C network to damp the oscillations to an acceptable level.

---

* General Electric - Ordnance Systems, Electronics Systems Division, Pittsfield, MA 01201.

** Environment-One Corporation, Schenectady NY 12301.

*** Gardner Associates, Schenectady, NY 12301.

Once all sample and return lines have been connected, the equipment is turned on and allowed to warm up. The flows are then adjusted to the proper rates. If the GE model is used, the vacuum gage on the front panel should read 8 inches of Hg as recommended in the operation manual. This gives a nominal flow rate of 6 ℓpm and a sample supersaturation after expansion of approximately 400%. The E-1 may be adjusted to any desired flow rate between about 0.6 ℓpm and 4.2 ℓpm.

The water supplies for the humidifiers are filled with a mixture of distilled water and a wetting agent. About 0.5% photo-flow (ethylene glycol) is used in the water. This reduces the surface tension of the water and allows the wick to wet better and more quickly.

Before any data can be taken the system should be leak checked. This is done by connecting all the D.B.'s in series, clamping one end off and pumping several inches of mercury vacuum. If the vacuum holds, data can be taken. If there is a leak it can be isolated by repeating the process with successively fewer D.B.'s until the leaky one is found. The exact location of the leak can be determined by putting a concentrated condensation nuclei source (e.g., a burning string) close to various parts of the D.B. When the leak is found, the CNC panel meter will rapidly rise.

There are two methods of data collection: graphing the CNC output on a strip chart recorder or directly writing down the meter reading. In general, a combination of the two is used to insure that no faulty connection exists between the CNC and the chart recorder or that the chart recorder is malfunctioning.

Since with either the GE or the E-1 CNC the largest flow possible is 6 ℓpm, a certain minimum amount of time is required to pull the sample through the parallel plate D.B.'s. If graphical techniques are used a characteristic output will be observed. The indicated concentration is zero while the clean air already in the D.B.'s is being exhausted. Then the output rises to a peak and stabilizes. It is at this point that meaningful data is being taken. However, if data is taken by meter readings alone this characteristic response is very difficult to follow. Adequate time must be allowed for transport through the D.B.'s before each reading is taken. These transport times have been calculated for 6 ℓpm for two types of parallel plate D.B.'s used: Thirty seconds for the 13 channel parallel plate and five minutes for each 98 channel parallel plate D.B. used. Quite frequently the in-stack concentrations are unsteady and data is normally recorded for several minutes after the system has had time to stabilize as a double check on the validity of the data and to provide some time averaging of the data on fluctuating sources. With the screen type diffusion batteries, transport times of about 1/10 of the times given above can be used.

If the GE CNC is used, diffusional sizing cut points are selected by changing the number of D.B.'s, since the flow rate is constant. However, with the E-1, the cut sizes may be adjusted by either changing the number of D.B.'s or the flow rate. The flow rate is sometimes held constant because source fluctuations introduce so much uncertainty that attempts to achieve high resolution are futile.

In order to obtain a set of data, the CNC is first connected directly to the diluter to obtain total concentration. Data are then taken by pulling the sample next through a single battery, two batteries, three batteries, etc., until all the available permutations of geometry and flow rate have been used.

If only one set of equipment is available, after data are collected at the inlet the equipment is carried to the outlet, set up, leak checked, and the above procedure is repeated.

### D.2.4 Data Reduction Techniques and a Sample Calculation

Fuchs et al (1962) presented a technique for calculating the particle size distribution from raw data, assuming that the size distribution was log normal. A technique suggested by Sinclair (1972) does not include this restriction. In Sinclair's method a nomograph is prepared using the penetration for each diffusion battery geometry and flow rate and a large number of monodisperse particle sizes. Comparing this nomograph with experimental penetrations, one calculates the particle size distribution using a "graphical stripping" process. However, it is usually more convenient to use a "$D_{50}$" technique like that used for the reduction of cascade impactor data.

Data for a given test condition are averaged as follows to yield a representation of the source at that condition. All instrument readings are converted to indicated concentrations by means of individual instrument calibration curves. These values are then corrected for dilution to obtain flue gas concentration. Since the CNC calibration curve is non-linear, multiplying by the dilution factor before converting to concentration will yield erroneous results. Next, process averaging is accomplished by taking appropriately weighted averages of the data obtained through the various process cycles. These weighted averages are found for each D.B. arrangement, including no D.B.'s, and test condition, both inlet and outlet.

For the sample calculation it is assumed that five parallel plate diffusion batteries were used, in four configurations, and at three flow rates to obtain data at twelve $D_{50}$ sizes. These five diffusion batteries consisted of one 12 channel (Type A) and four 98 channel (Type B) units. The four sampling configurations were (1) one Type A, (2) one Type B, (3) two Type B in series, and (4) four Type B in series. These diffusion batteries are similar to the one depicted in Figure D7. The three flow rates were 1, 6, and 10 liters per minute. The aerosol was sampled from a Sample Extraction and Dilution System and the total number of particles entering and exiting the diffusion batteries was determined using a condensation nuclei counter (CNC). The sample data for this experiment is shown in Table D1.

To calculate the particle size for 50% penetration (the $D_{50}$) through a diffusion battery configuration, the following equations must be used.

The penetration of a rectangular plate diffusion battery is given by

$$P = \frac{n}{n_0} = \Big[0.9104e^{-1.88522DfY} + 0.0531e^{-21.431DfY} + 0.015e^{-62.317DfY} + 0.0068e^{-124.537DfY}\Big]^m \qquad \text{(D1)}$$

where Df = Particle diffusivity, cm /sec,

Y = Diffusion battery flow rate-geometry constant, sec/cm ,

m = Number of identical batteries in series,

$n_0$ = Diffusion battery inlet concentration, #/cm$^3$, and

n = Diffusion battery outlet concentration, #/cm$^3$.

The particle diffusivity, Df, is given by

$$Df = kTB, \qquad \text{(D2)}$$

where k = Boltzmann's Constant, gm cm$^2$/sec$^2$ °K,

T = Absolute Temperature, °K, and

B = Particle Mechanical Mobility, sec/gm.

# TABLE D1

# DIFFUSION BATTERY SAMPLE CALCULATION DATA

# CNC

| Configuration | Flow Rate (ℓ/min) | Calc. $D_{50}$ (μm) | Reading (#/cm$^3$) | Dilution Factor | Actual Concentration (#/cm$^3$ x 10$^6$) |
|---|---|---|---|---|---|
| 1 Type A | 6 | 0.02 | 53000 | 1000 | 53.0 |
| 1 Type B | 6 | 0.06 | 43000 | 500 | 21.5 |
| 2 Type B | 6 | 0.098 | 45000 | 200 | 9.0 |
| 4 Type B | 6 | 0.17 | 11000 | 200 | 2.2 |
| 1 Type A | 1 | 0.028 | 84000 | 500 | 42.0 |
| 1 Type B | 1 | 0.07 | 33600 | 500 | 16.8 |
| 2 Type B | 1 | 0.12 | 29000 | 200 | 5.8 |
| 4 Type B | 1 | 0.20 | 5500 | 200 | 1.1 |
| 1 Type A | 10 | 0.015 | 64000 | 1000 | 64.0 |
| 1 Type B | 10 | 0.045 | 62000 | 500 | 31.0 |
| 2 Type B | 10 | 0.08 | 66500 | 200 | 13.3 |
| 4 Type B | 10 | 0.14 | 20500 | 200 | 4.1 |

The particle mechanical mobility is given by

$$B = \left[1 + 2.49\ (\lambda/d) + 0.84\ (\lambda/d)e^{-(0.44)(d/\lambda)}\right]/3\pi\mu d \qquad \text{(D3)}$$

where $\lambda$ = gas mean free path, cm,
$d$ = particle diameter, cm, and
$\mu$ = gas viscosity, gm/cm-sec.

The gas mean free path is given by

$$\lambda = 3.109 \times 10^{-1}\frac{\mu}{P}\sqrt{T/M} \qquad \text{(D4)}$$

where $P$ = Ambient pressure, atm
$T$ = Absolute temperature, °K, and
$M$ = gas mean molecular weight, gm/gm-mole.

The approximate gas viscosity is given by

$$\mu = (0.494\ (T-294°K) + 181) \times 10^{-6}\ \text{gm/cm-sec.} \qquad \text{(D5)}$$

The diffusion battery flow rate-geometry constant, Y, is given by

$$Y = \frac{4LhN}{QW} \qquad \text{(D6)}$$

where $L$ = Channel length, cm,
$h$ = Channel height, cm,
$N$ = Number of channels,
$Q$ = Flow rate, $cm^3$/sec, and
$W$ = Channel width, cm.

See Figure D7.

The two diffusion batteries under consideration in this example have the following dimensions.

| | Type A | Type B |
|---|---|---|
| L | 45.72 cm | 45.72 cm |
| h | 10.15 cm | 11.46 cm |
| W | 0.10 cm | 0.10 cm |
| N | 13 | 98 |

Thus, $Y_A = 2.358 \times 10^3$ sec/cm$^2$ and $Y_B = 2.006 \times 10^4$ sec/cm$^2$ for $Q = 6$ ℓ/min.

The manipulation of these equations allows one to calculate the penetration of the diffusion batteries at different flow rate and particle-size combinations. After plotting the penetration versus particle size, the $D_{50}$'s can be determined.

To aid in the calculation of the particle diffusivity and penetration of a particular diffusion battery arrangement, two programs have been written for the Hewlett-Packard HP-65 Programmable Calculator. If a programmable calculator is not available, the calculations can be done manually using the equations given above. The calculator programs and their applications are described below:

Program 1 is used to calculate the viscosity ($\mu$) and mean free path ($\lambda$) of standard air. These values are then used to calculate the diffusivity (Df) for a monodisperse aerosol having diameter d. Given values for the flow rate-geometry configuration (Y) and the diffusivity (Df), Program 2 is used to calculate the theoretical penetration ($n/n_0$).

## Program 1: Diffusivity (Program Steps are Listed in Table D2.)

Over the temperature range from 0-350°C, the viscosity of dry standard air is very nearly linear. For a given temperature $\mu$ can be found, in CGS units, from:

$$\mu = \left[0.494\ (T - 294°K) + 181\right] \times 10^{-6} \text{ poise}$$

Knowing the viscosity, the mean free path is given by

$\lambda = 5.77 \frac{\mu}{P} \sqrt{T} \times 10^{-2}$ cm for standard air (P is the absolute pressure, atm.).

From a knowledge of $\mu$ and $\lambda$ for the carrier gas, the diffusivity ($Df_i$) of a monodisperse aerosol having diameter ($d_i$) is given by the following equation:

$$Df_i = \left(1.46 \times 10^{-17}\right) \frac{T}{\mu d_i} \left[1 + 2.49\left(\frac{\lambda}{d_i}\right) + 0.84\left(\frac{\lambda}{d_i}\right) e^{-(0.44)(d_i/\lambda)}\right]$$

for T in °K, $\mu$ in poise, $\lambda$ and $d_i$ in cm.

User Instructions. Enter the program shown in Table D2.

To calculate $\mu$ and $\lambda$ for Standard Air having $250°K < T < 600°K$:

1. Load storage registers with the following variables

| | | | |
|---|---|---|---|
| Temperature | T, | °K, | STO 2 |
| Absolute Pressure, | P, | "Hg, | STO 3 |

2. Start program "$\mu$, $\lambda$" "B"

## TABLE D2

## PROGRAM 1 - PARTICLE DIFFUSIVITY (Df)
## HP-65 CALCULATOR PROGRAM FOR A PARALLEL PLATE DIFFUSION BATTERY

| CODE | KEYS |
|---|---|
| LBL | 23 |
| A | 11 |
| RCL 1 | 34 01 |
| RCL 5 | 34 05 |
| ÷ | 81 |
| · | 83 |
| 4 | 04 |
| 3 | 03 |
| 5 | 05 |
| CHS | 42 |
| X | 71 |
| $f^{-1}$ | 32 |
| LN | 07 |
| RCL 5 | 34 05 |
| RCL 1 | 34 01 |
| ÷ | 81 |
| STO 8 | 33 08 |
| X | 71 |
| · | 83 |
| 8 | 08 |
| 4 | 04 |
| X | 71 |
| 1 | 01 |
| + | 61 |
| RCL 8 | 34 08 |
| 2 | 02 |
| · | 83 |
| 4 | 04 |
| 9 | 09 |
| 2 | 02 |
| X | 71 |
| + | 61 |
| RCL 2 | 34 02 |
| X | 71 |
| 1 | 01 |
| 4 | 04 |
| 6 | 06 |
| EEX | 43 |
| CHS | 42 |
| 1 | 01 |
| 9 | 09 |
| X | 71 |
| RCL 4 | 34 04 |
| ÷ | 81 |
| RCL 1 | 34 01 |
| ÷ | 81 |
| STO 7 | 33 07 |
| RTN | 24 |
| LBL | 23 |
| B | 12 |
| · | 83 |
| 4 | 04 |
| 9 | 09 |
| 5 | 05 |
| RCL 2 | 34 02 |
| 2 | 02 |
| 9 | 09 |
| 4 | 04 |
| − | 51 |
| X | 71 |
| 1 | 01 |
| 8 | 08 |
| 2 | 02 |
| + | 61 |
| EEX | 43 |
| CHS | 42 |
| 6 | 06 |
| X | 71 |
| STO 4 | 33 04 |
| RCL 3 | 34 03 |
| ÷ | 81 |
| RCL 2 | 34 02 |
| f | 31 |
| √ | 09 |
| X | 71 |
| · | 83 |
| 0 | 00 |
| 5 | 05 |
| 7 | 07 |
| 7 | 07 |
| X | 71 |
| STO 5 | 33 05 |
| RTN | 24 |

| | | | | | |
|---|---|---|---|---|---|
| $R_1$ | d | $R_4$ | $\mu$ | $R_7$ | $Df_i$ |
| $R_2$ | T | $R_5$ | $\lambda$ | $R_8$ | (WORK) |
| $R_3$ | P | $R_6$ | (BLANK) | $R_9$ | (BLANK) |

3. Output:

Values of $\mu$ are stored in the correct storage register for retrieval during calculation of Df

| | |
|---|---|
| To display $\mu$ (units of poise) | RCL 4 |
| To display $\lambda$ (units of cm) | RCL 5 |

To calculate $Df_i$ for a monodisperse aerosol

4. Load Storage registers with the following variables

| | | | | |
|---|---|---|---|---|
| Temperature, | T, | °K, | STO 2 | Performed in Steps 1 and 2 |
| Viscosity, | $\mu$, | poise, | STO 4 | |
| Mean Free Path, | $\lambda$, | cm, | STO 5 | |
| Diameter of the monodisperse aerosol, | $d_i$, | cm, | STO 1 | |

5. Start program "Diff" "A"

6. Output:

Displayed value is "Df" in cgs units (also stored in register 7).

Test Problem

Find $\mu$, $\lambda$, and $Df_i$ for T = 75.2°F, P = 14.39 PSIA, and d = 0.023 microns ($\mu$m).

1. Convert T, P, and $d_i$ to the proper units then store in registers 2, 3, and 1, respectively.

75.2°F = 24°C = 297°K, store in register 2

$14.39 \text{ PSIA} \times 2.036 \frac{\text{in. Hg}}{\text{PSIA}} = 29.30$ in. Hg, store in register 3

$0.023\ \mu m = 0.023 \times 10^{-4}$ cm $= 2.3 \times 10^{-6}$ cm, store in register 1

2. Start Program B. $\lambda$ is displayed ($\lambda = 6.23 \times 10^{-6}$ cm), RCL 4 to display ($\mu = 1.83 \times 10^{-4}$ poise).

3. Start Program A. $Df_i$ is displayed ($Df_i = 9.95 \times 10^{-5} \text{cm}^2/\text{sec}$).

Program 2: Theoretical Penetration (Program Steps are Listed in Table D3.)

When the diffusivity ($Df_i$) of a monodisperse aerosol of diameter $d_i$ is known, and the flow rate geometry configuration ($Y_j$) for the diffusional apparatus (diffusion battery) are known, fractional penetration as a function of size ($d_i$) is given by

$$p_{i,j} = 0.9104e^{-1.8852Df_iY_j} + 0.0531e^{-21.431Df_iY_j}$$

$$+ 0.015^{-62.317Df_iY_j} + 0.068e^{-124.537Df_iY_j}$$

If several diffusional configurations are used in series where the aerosol is allowed to remix before going through the next configuration, the final penetration is the product of the penetration for each, thus

$$p_i = \prod_j p_{i,j}$$

If m diffusion batteries having the same $Y_j$ value are used in series, then

$$p_i = \left(p_{i,j}\right)^m$$

TABLE D3

PROGRAM 2 - PARTICLE PENETRATION (p)
HP-65 CALCULATOR PROGRAM FOR A PARALLEL PLATE DIFFUSION BATTERY

| CODE | KEYS |
|---|---|
| LBL | 24 |
| A | 11 |
| RCL 7 | 34 07 |
| RCL 6 | 34 06 |
| X | 71 |
| STD 8 | 33 08 |
| 1 | 01 |
| · | 83 |
| 8 | 08 |
| 8 | 08 |
| 5 | 05 |
| 2 | 02 |
| CHS | 42 |
| X | 71 |
| $f^{-1}$ | 32 |
| LN | 07 |
| · | 83 |
| 9 | 09 |
| 1 | 01 |
| 0 | 00 |
| 4 | 04 |
| X | 71 |
| RCL 8 | 34 08 |
| 2 | 02 |
| 1 | 01 |
| · | 83 |
| 4 | 04 |
| 3 | 03 |
| 1 | 01 |
| CHS | 42 |
| X | 71 |
| $f^{-1}$ | 32 |
| LN | 07 |
| · | 83 |
| 0 | 00 |
| 5 | 05 |
| 3 | 03 |
| 1 | 01 |
| X | 71 |
| + | 61 |
| RCL 8 | 34 08 |
| 6 | 06 |
| 2 | 02 |
| · | 83 |
| 3 | 03 |
| 1 | 01 |
| 7 | 07 |
| CHS | 42 |
| X | 71 |
| $f^{-1}$ | 32 |
| LN | 07 |
| · | 83 |
| 0 | 00 |
| 1 | 01 |
| 5 | 05 |
| 3 | 03 |
| X | 71 |
| + | 61 |
| RCL 8 | 34 08 |
| 1 | 01 |
| 2 | 02 |
| 4 | 04 |
| · | 83 |
| 5 | 05 |
| 3 | 03 |
| 7 | 07 |
| CHS | 42 |
| X | 71 |
| $f^{-1}$ | 32 |
| LN | 07 |
| · | 83 |
| 0 | 00 |
| 0 | 00 |
| 6 | 06 |
| 8 | 08 |
| X | 71 |
| + | 61 |
| RCL | 34 |
| 9 | 09 |
| g | 35 |
| $y^x$ | 05 |
| RTN | 24 |

| | | | | | |
|---|---|---|---|---|---|
| $R_1$ | (BLANK) | $R_4$ | (BLANK) | $R_7$ | $Df_i$ |
| $R_2$ | (BLANK) | $R_5$ | (BLANK) | $R_8$ | (WORK) |
| $R_3$ | P | $R_6$ | $Y_j$ | $R_9$ | m |

To calculate $p_{i,j}$ for m identical batteries in series, enter program in Table D3.

1. Load storage registers with the collowing variables

| | | | |
|---|---|---|---|
| Diffusivity, | $Df_i$, | cgs, units, | STO 7 |
| Flow rate-geometry configuration | $Y_j$, | cgs units, | STO 6 |
| Number of identical batteries in series | m, | integer, | STO 9 |

2. Start Program "A"

Displayed value is the fractional penetration ($p_{i,j}$) of this monodisperse aerosol of size $d_i$ through m identical diffusion batteries in series.

Test Problems

1. Load data

$Y_j = 2.36 \times 10^3$ in cgs units STO 6

$Df_i = 5.39 \times 10^{-6}$ in cgs units STO 7

$m = 1$ STO 9

2. Calculate p "A"

$p = 9.38 \times 10^1 = 93.8\%$

Therefore, the penetration of the diffusion battery Type A at a flow rate of 6 liters/minute is 93.8% for 0.023 μm particles.

After calculating the penetration, a graph similar to Figure D9 will be obtained. The $D_{50}$'s can be determined from this graph as indicated. The experimental data for this sample calculation are shown in Figure D11.

A cumulative particle size distribution is plotted using the corrected concentration from the last column in Table D1 as the ordinate and the $D_{50}$'s for each configuration as the abscissa. Differential number graphs are obtained by differentiation of the cumulative curve (finite differences) as described in Sections C.8 and C.9.

## D.3 SUBMICRON PARTICLE SIZING TECHNIQUES USING THE ELECTRICAL MOBILITY PRINCIPLE

### D.3.1 Electrical Mobility

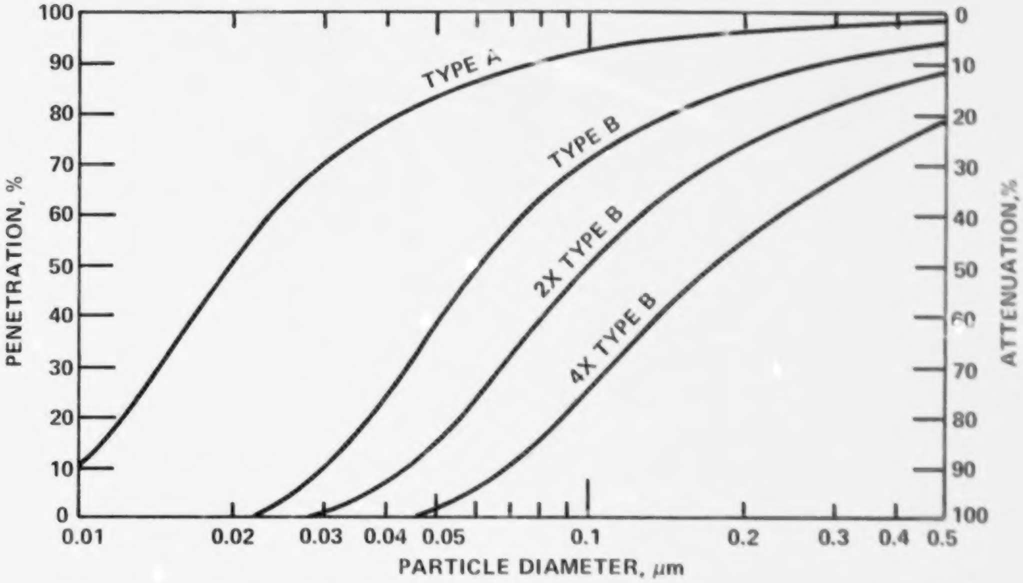
If a particle charge is known, measurements of the electrical mobility can be used to determine the particle size. This concept has been used by Liu, Whitby, and Pui (1974) at the University of Minnesota to develop a series of Electrical Aerosol Analyzers (EAA). A schematic of this device is shown in Figure D10. The EAA has the distinct advantage of very rapid data acquisition compared to parallel plate diffusion batteries used with condensation nuclei counters (two minutes as opposed to two hours for a single size distribution analysis).

### D.3.2 Thermosystems Model 3030 Electrical Aerosol Size Analyzer*

The EAA is designed to size particles in the range of 0.0032 micrometer diameter to 1.0 micrometer diameter. It can size solids

---

* Thermosystems, Inc., St. Paul, MN 55113.



3630 042

*Figure D9. Theoretical parallel plate diffusion battery penetration curves.*

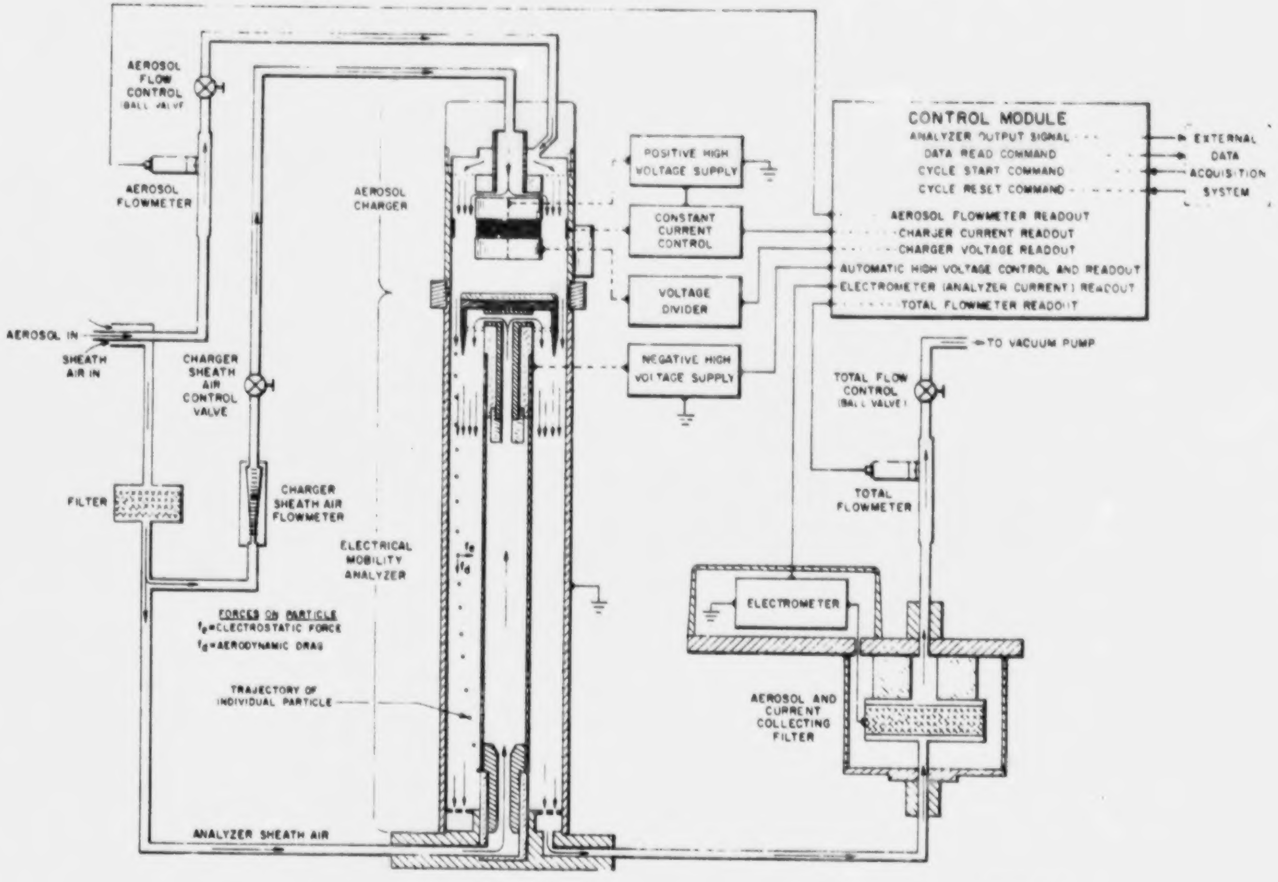

AEROSOL FLOW CONTROL (BALL VALVE)
AEROSOL FLOWMETER
AEROSOL IN
SHEATH AIR IN
CHARGER SHEATH AIR CONTROL VALVE
FILTER
CHARGER SHEATH AIR FLOWMETER
AEROSOL CHARGER
ELECTRICAL MOBILITY ANALYZER
FORCES ON PARTICLE
$f_e$=ELECTROSTATIC FORCE
$f_d$=AERODYNAMIC DRAG
$f_e$
$f_d$
TRAJECTORY OF INDIVIDUAL PARTICLE
ANALYZER SHEATH AIR
POSITIVE HIGH VOLTAGE SUPPLY
CONSTANT CURRENT CONTROL
VOLTAGE DIVIDER
NEGATIVE HIGH VOLTAGE SUPPLY
CONTROL MODULE
ANALYZER OUTPUT SIGNAL
DATA READ COMMAND
CYCLE START COMMAND
CYCLE RESET COMMAND
AEROSOL FLOWMETER READOUT
CHARGER CURRENT READOUT
CHARGER VOLTAGE READOUT
AUTOMATIC HIGH VOLTAGE CONTROL AND READOUT
ELECTROMETER (ANALYZER CURRENT) READOUT
TOTAL FLOWMETER READOUT
EXTERNAL DATA ACQUISITION SYSTEM
TO VACUUM PUMP
TOTAL FLOW CONTROL (BALL VALVE)
TOTAL FLOWMETER
ELECTROMETER
AEROSOL AND CURRENT COLLECTING FILTER
3630-043

*Figure D10. Schematic diagram of the electrical aerosol analyser. After Liu and Pui(1975).*

and non-volatile liquids. The instrument's concentration range of 1 to 1000 μg/m$^3$ requires that a sample extraction-dilution system (as described earlier) be used.

The EAA is operated in the following manner. As a vacuum pump draws the aerosol through the analyzer (See Figure D10), a positive corona (generated at a high voltage wire within the charging section) gives the sample a positive electrical charge.

The charged aerosol flows from the charger to the analyzer section as an annular cylinder of aerosol surrounding a cone of clean air. A metal rod, to which a variable, negative voltage can be applied, passes axially through the center of the analyzer tube. Particles smaller than a certain size (with highest electrical mobility) are drawn to the collecting rod when the voltage corresponding to that size is on the rod. Larger particles pass through the analyzer tube and are collected by a filter. The electrical charges on these particles drain off through an electrometer, giving a measure of current.

A step increase in rod voltage will cause particles of a larger size to be collected by the rod with a resulting decrease in electrometer current. This decrease in current can be related to the increased number of collected particles by the current sensitivities ($\Delta N/\Delta I$) as determined by Liu and Pui (1975). Liu's work determined empirical values for the current sensitivities for the case when the ion electrical mobility had a value of 1.4 cm$^3$/volt-sec. Marlow, Reist, and Dwiggins (1976) have done work that indicates that the presence of certain trace elements (e.g., 0.01 ppm $SO_2$) in the instrument's sheath air can significantly affect data obtained with the EAA. They suggested several possible explanations, one of which was that any $SO_2$, present in the charger sheath air, can contribute substantially to the current carrying ion species, and thus, significantly alter the effective ion mobility for particle charging. A total of eleven voltage steps divide the 0.0032 to 1.0 micron size range of the instrument into ten equal logarithmic size intervals. Different size intervals can be programmed via an optional plug-in memory card.

The electrical aerosol analyzer can be operated either automatically or manually. In the automatic mode, the analyzer steps through the entire size range. For size and concentration monitoring over an extended period of time, the analyzer may be intermittently triggered by an external timer. The standard readout consists of a digital display within the control circuit module, although a chart recorder output is available. It is almost always advantageous to use a strip chart recorder to record the data. This allows the operator to identify a stable reading superimposed on source variations and gives a permanent record of the raw data.

### D.3.3 Using the TSI Model 3030 Electrical Aerosol Analyzer With a Sample Extraction-Dilution System To Measure Concentrations of Submicron Aerosols in Industrial Flue Gases

Once the equipment is set up as shown in Figure D3, the flows are adjusted through the sample orifice and the dilution air orifice, to obtain the desired dilution factor. The EAA is placed in a manual mode and the current readings for each channel are recorded with a strip chart recorder. Manual control allows run times of from two to five minutes in each of the nine channels. This allows one to average out rapid source fluctuations. At the beginning of each day, the internal calibration points and flows through the EAA are checked, as described in the instrument manual. In order to assure correct residence time through the charger section the operator must measure the temperature and pressure of the aerosol in order to calculate the mass flows that yield actual flows of 4 ℓpm and 50 ℓpm.

### D.3.4 Data Reduction Techniques and a Sample Calculation

It is assumed that a Thermo-Systems Inc. Model 3030 Electrical Aerosol Size Analyzer (EAA) with a 0.0032 μm to 0.360 μm range at the normal operating conditions has been used to determine concentration vs size information in the ultrafine size range for the effluent of a fabric filter. The EAA sampled the gas stream after the sample was extracted with a Sample Extraction and Dilution System as described in Section D.1.7.

The EAA was placed in a manual scan mode and the current readings for each channel were recorded with a strip chart recorder. Manual control allowed run times of up to one minute for each of the nine channels. This allowed the averaging of rapid source fluctuations.

The theory of operation and basic equations for the EAA have been given by Sem (1975). The EAA was initially marketed in a configuration having an $N_t$ product of $7 \times 10^6$ (ions/cm)(sec) and a set of preprogrammed collection rod voltages aimed at obtaining eleven size cuts between 0.0032 and 1.00 μm. Calibration by Liu and Pui (1975) showed that for an $N_t$ product of $7 \times 10^6$ [and an assumed ion electrical mobility of 1.4 cm$^3$/(volt-sec)] these preprogrammed voltages resulted in eleven size cuts between 0.032 and 0.36 μm. Liu's work determined the correct size cuts and associated current sensitivities for the preprogrammed voltages. It is these values that are shown in Table D4 and used in the example. Liu's work also determined an $N_t$ value and set of collection rod voltages that obtained the originally desired set of size cuts (from 0.0032 to 1.00 μm). This $N_t = 1 \times 10^7$ configuration and the set of voltages used by Liu are now the standard configuration for the Thermo System Inc. EAA. The mechanics of the data reduction for the $N_t = 1 \times 10^7$ configuration and the $N_t = 7 \times 10^6$ configuration (shown in the example herein) are identical. A computerized data reduction program for the $N_t = 1 \times 10^7$ configuration (Liu and Kapodia, 1977) is now commercially available from Thermo Systems Inc. This program attempts to correct for the multiple charging effects noted by Liu by assuming a log normal distribution (unimodel or bimodel) and using Liu's calibration matrix to arrive at a best fit to the measured set of I values.

Table D4 is essentially self-explanatory. The heading "$D_p$, μm" (Column 3) is the particle diameter in micrometers. A value of 0.100 μm means that particles equal to or smaller than this diameter are collected in the analyzer tube while larger particles penetrate to the current collecting filter where an electrometer measures the total current carried by the unprecipitated particles. This current represents the charges on all particles larger than 0.100 μm. This measured current is the basic output of the Model 3030.

The fourth column ($D_{pi}$, μm) is the geometric mean diameter of the particles represented by the current difference of two successive steps (Channel No.'s). For example, the difference in current for the 0.100 μm cut-off and the current for the 0.0178 μm cut-off is the total current collected from particles between these sizes, or rather for a mean diameter of 0.0133 μm. The current differences are entered in Column 8 headed "I, pA" (picoAmps).

The fifth column gives the revised calibration factor (based on the calibration by Liu and Pui (1975)) for each of the eight size bands. These factors are in units of particles per cm per picoAmpere. Multiplying this size specific current sensitivity, $\Delta N/\Delta I$, (Column 5) by the current difference, $\Delta I$, (Column 8) gives

## TABLE D4

EAA (Model 3030) Data Reduction Form
Concentration, Cumulative Concentration, and $\Delta N_s/\Delta \mathrm{LogD}$ From
Scan No. ___ For DF = ____
Nt = $7\mathrm{x}10^5$

| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Channel No. | Collector Voltage | $D_p$, µm | $D_{pi}$, µm | $\Delta N/\Delta I$ | $\Delta \log D_p$ | I,pA | ΔI,pA | ΔN | $\Delta N_s$ | $\Sigma N_s$ | $\Delta N_s/\Delta \log D$ |
| 3 | 196 | 0.0100 | | | | | | | | | |
| | | | 0.0133 | $4.76\mathrm{x}10^5$ | 0.250 | | | | | | |
| 4 | 593 | 0.0178 | | | | | | | | | |
| | | | 0.0215 | $2.33\mathrm{x}10^5$ | 0.165 | | | | | | |
| 5 | 1220 | 0.026 | | | | | | | | | |
| | | | 0.0306 | $1.47\mathrm{x}10^5$ | 0.141 | | | | | | |
| 6 | 2183 | 0.036 | | | | | | | | | |
| | | | 0.0502 | $8.33\mathrm{x}10^4$ | 0.289 | | | | | | |
| 7 | 3515 | 0.070 | | | | | | | | | |
| | | | 0.0917 | $4.26\mathrm{x}10^4$ | 0.234 | | | | | | |
| 8 | 5387 | 0.120 | | | | | | | | | |
| | | | 0.149 | $2.47\mathrm{x}10^4$ | 0.188 | | | | | | |
| 9 | 7152 | 0.185 | | | | | | | | | |
| | | | 0.219 | $1.56\mathrm{x}10^4$ | 0.148 | | | | | | |
| 10 | 8642 | 0.260 | | | | | | | | | |
| | | | 0.306 | $1.10\mathrm{x}10^4$ | 0.141 | | | | | | |
| 11 | 9647 | 0.360 | | | | | | | | | |

the total number of particles, $\Delta N$, (Column 9) in units of particles per $cm^3$, within this size band (Column 4) for the diluted aerosol. To correct for dilution and find in-stack concentrations, multiply Column 9 by the dilution factor (DF) and enter the result, $\Delta N_s$, in Column 10. Columns 6 and 12 are used for $\Delta N_s/\Delta \mathrm{Log}D$ information calculated from the number distribution in Column 10. Column 11 is used for cumulative concentrations, corrected for dilution. All concentration have been corrected to engineering standard (normal) conditions by the dilution factor. Engineering standard or normal conditions are defined as dry gas at 21°C and 760 mm Hg pressure.

The basic data from the EAA is cumulative current for each of nine channels (Column 7). One must then take the differences of the current readings for successive channels (Column 8) in order to find $\Delta N$, etc. The $\Delta I$ values are multiplied by a series of constants ($\Delta N/\Delta I_i$, $DF_j$) to arrive at $\Delta N_s$ (concentration in stack corrected to dry, standard conditions). While a single scan should be made at a constant dilution, different scans may be made at different dilutions. To simplify the arithmetic for each test condition, the product $\alpha_i = \Delta I_{i,j} \times DF_j$ is formed and all such inlet (outlet) products for the same size band are averaged.

## SUMMARY OF THE CALCULATION FORMAT

### STEP 1

A. Calculate the average instrument reading (I) for each channel as obtained from the strip chart recording of channel current vs. time.

B. Calculate all dilution factors ($DF_j$).

STEP 2

Calculate current differences ($\Delta I_{i,j}$) from adjacent channels and average the $\alpha_i$ products ($\alpha_i = \Delta I_{i,j} \times DF_j$) for the same size band for all scans taken for the same test conditions. Calculate 90% confidence intervals for each $\bar{\alpha}_i$. Note: the i subscript denotes size and the j subscript denotes dilution setting.

STEP 3

Using $\bar{\alpha}_i$ and Table D6 calculate "number concentration" ($\Delta N_s$), "average cumulative concentration of all particles having diameter greater than the indicated size" $\Sigma(\Delta N_s)$, and "$\Delta N_s/\Delta \mathrm{Log}D$" for each size band for each test condition.

STEP 4

Plot "Cumulative Concentration vs. Size" for each test condition.

STEP 5

Plot $\Delta N_s/\Delta \mathrm{Log}D$ (with upper and lower 90% confidence limits) vs. size for each test condition.

SAMPLE CALCULATION FOLLOWING THE CALCULATION FORMAT

Table D5 contains hypothetical test data for the following sample calculation.

STEP 1

A. Calculate the average instrument reading (I) for each channel as obtained from the strip chart recording of channel current vs. time. Each complete size scan (Table D5) consists of nine

# TABLE D5

EAA Current Readings (I, in picoamps and Dilution Factors)
For This Sample Calculation: Hypothetical Inlet Data
$Nt = 7x10^6$

| SCAN | Time | CH 3 | CH 4 | CH 5 | CH 6 | CH 7 | CH 8 | CH 9 | CH 10 | CH 11 | Dilution Factor |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 1:30p | 2.869 | 2.734 | 2.519 | 2.227 | 1.362 | .682 | .242 | .102 | .020 | 255 |
| 2 | 1:32 | 2.835 | 2.711 | 2.495 | 2.205 | 1.344 | .669 | .220 | .075 | -.010 | 255 |
| 3 | 1:34 | 2.841 | 2.709 | 2.500 | 2.200 | 1.340 | .655 | .218 | .081 | .001 | 255 |
| 4 | 1:36 | 2.859 | 2.722 | 2.522 | 2.235 | 1.368 | .676 | .226 | .096 | .010 | 255 |
| 5 | 1:38 | 2.866 | 2.740 | 2.530 | 2.251 | 1.381 | .714 | .279 | .137 | .052 | 255 |
| 6 | 1:40 | 2.866 | 2.736 | 2.531 | 2.238 | 1.378 | .698 | .255 | .115 | .033 | 255 |
| 7 | 1:45 | 6.477 | 6.188 | 5.716 | 5.056 | 3.111 | 1.575 | .565 | .243 | .053 | 113 |
| 8 | 1:47 | 6.580 | 6.288 | 5.818 | 5.153 | 3.233 | 1.613 | .510 | .195 | .010 | 113 |
| 9 | 1:49 | 6.377 | 6.087 | 5.620 | 4.960 | 3.021 | 1.526 | .537 | .227 | .032 | 113 |
| 10 | 1:51 | 6.390 | 6.094 | 5.614 | 4.956 | 3.006 | 1.467 | .492 | .187 | .005 | 113 |

instrument readings (I, Column 7 of Table D4). These instrument readings are the average current outputs as taken from the strip chart recordings, for each of the nine channels. Run times were manually controlled and varied from two to ten minutes per channels 3, 4, 5, ..., 11. Table D5 gives the instrument readings used as data for the sample calculation (10 scans, 90 average current readings).

B. Calculate all dilution factors ($DF_j$; corrected to engineering standard (normal) conditions: 70°F (20°C) and 29.92 inches of mercury pressure (760 mm Hg)).

STEP 2

Calculate current differences ($\Delta I_{i,j}$) from adjacent channels and average the $\alpha_i$ products for the same size band for all scans taken at the same test condition. Calculate 90% confidence intervals for each $\bar{\alpha}_i$. (Refer to Section C.8.5).

$$\alpha_i = \Delta I_{i,j} \times DF_j$$

where i denotes the size band and j denotes the dilution value.

For channels 3-4 we have:

Scan #1: $\alpha_{3-4,1} = (0.135)(255)$ pA
#2: $\alpha_{3-4,1} = (0.124)(255)$ pA
#3: $\alpha_{3-4,1} = (0.132)(255)$ pA
.
.
.
#9: $\alpha_{3-4,2} = (0.290)(113)$ pA
#10: $\alpha_{3-4,2} = (0.296)(113)$ pA

thus, $\bar{\alpha}_{3-4} = 33.179$ pA; n = 10 and CI = 0.579.

In a similar manner we can find $\bar{\alpha}_{4-5}$, $\bar{\alpha}_{5-6}$, . . ., $\bar{\alpha}_{10-11}$.

Thus, the mean, with upper and lower 90% confidence limits for $\bar{\alpha}_{3-4}$ is given by:

$$\bar{\alpha}_{3-4} = (33.179 \pm 0.579) \text{ pA}$$

or

$$\bar{\alpha}_{3-4} = (33.2 \pm 0.6) \text{ pA}$$

STEP 3

Using $\bar{\alpha}_i$ and Table D6 calculate "number concentration" ($\Delta N_S$), "average cumulative concentration . . ." ($\Sigma\Delta N_S$), and "$\Delta N_S/\Delta \text{Log} D$" for each size band for each test condition.

Table D7 shows these calculations for the sample data of Table D5. Column 7 is $\bar{\alpha}$ as shown in Step 2. Column 8 is the product of columns 7 and 5. Column 9 is the summation of 8 for all sizes "equal to or greater than the indicated size." Column 10 is column 5 times column 7 divided by column 6.

STEP 4

Plot cumulative concentration vs. size for each test conditions. For the sample data set of Table D5 this would be the concentrations in Table D7 column 9 plotted against the sizes in column 4. No errors bars are used.

STEP 5

Plot $\Delta N_S/\Delta \text{Log} D$ with upper and lower 90% confidence limits for each test condition.

# TABLE D6

EAA (Model 3030) Data Reduction Form
Concentration, Cumulative Concentration, and $\Delta N_s/\Delta \text{LogD}$
From Average $\bar{\alpha}$ For Condition ______
$Nt = 7\text{x}10^6$

| 1<br>Channel No. | 2<br>Collector Voltage | 3<br>$D_p$, µm | 4<br>$D_{pi}$, µm | 5<br>$\Delta N/\Delta I$ | 6<br>$\Delta \log D_p$ | 7<br>$\bar{\alpha}$ | 8<br>$\Delta N_s$ | 9<br>$\Sigma \Delta N_s$ | 10<br>$\Delta N_s/\Delta \log D$ |
|---|---|---|---|---|---|---|---|---|---|
| 3 | 196 | 0.0100 | | | | | | | |
| | | | 0.0133 | $4.76\text{x}10^5$ | 0.250 | | | | |
| 4 | 593 | 0.0178 | | | | | | | |
| | | | 0.0215 | $2.33\text{x}10^5$ | 0.165 | | | | |
| 5 | 1220 | 0.026 | | | | | | | |
| | | | 0.0306 | $1.47\text{x}10^5$ | 0.141 | | | | |
| 6 | 2183 | 0.036 | | | | | | | |
| | | | 0.0502 | $8.33\text{x}10^4$ | 0.289 | | | | |
| 7 | 3515 | 0.070 | | | | | | | |
| | | | 0.0917 | $4.26\text{x}10^4$ | 0.234 | | | | |
| 8 | 5387 | 0.120 | | | | | | | |
| | | | 0.149 | $2.47\text{x}10^4$ | 0.188 | | | | |
| 9 | 7152 | 0.185 | | | | | | | |
| | | | 0.219 | $1.56\text{x}10^4$ | 0.148 | | | | |
| 10 | 8642 | 0.260 | | | | | | | |
| | | | 0.306 | $1.10\text{x}10^4$ | 0.141 | | | | |
| 11 | 9647 | 0.360 | | | | | | | |

## TABLE D7

EAA (Model 3030) Data Reduction Form
Concentration, Cumulative Concentration, and $\Delta N_s/\Delta \mathrm{LogD}$
From Average ΔI For Condition Inlet
(Sample Calculation)
$Nt = 7x10^6$

| 1<br>Channel No. | 2<br>Collector Voltage | 3<br>$D_p$, μm | 4<br>$D_{pi}$, μm | 5<br>$\Delta N/\Delta I$ | 6<br>$\Delta \log D_p$ | 7<br>$\bar{\alpha}$ | 8<br>$\Delta N_s$ | 9<br>$\Sigma\Delta N_s$ | 10<br>$\Delta N_s/\Delta \mathrm{LogD}$ |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | $x10^6$ | $x10^6$ | $x10^6$ |
| 3 | 196 | 0.0100 | | | | | | | |
| | | | 0.0133 | $4.76x10^5$ | 0.250 | 33.2±.6 | 15.8±.3 | 68.4 | 63.2±1.1 |
| 4 | 593 | 0.0178 | | | | | | | |
| | | | 0.0215 | $2.33x10^5$ | 0.165 | 53.3±.7 | 12.4±.2 | 52.6 | 75.3±1.0 |
| 5 | 1220 | 0.026 | | | | | | | |
| | | | 0.0306 | $1.47x10^5$ | 0.141 | 74.3±.8 | 10.9±.1 | 40.2 | 7[illegible].8 |
| 6 | 2183 | 0.036 | | | | | | | |
| | | | 0.0502 | $8.33x10^4$ | 0.289 | 219.8±.8 | 18.3±.1 | 29.3 | 63.4±.2 |
| 7 | 3515 | 0.070 | | | | | | | |
| | | | 0.0917 | $4.26x10^4$ | 0.234 | 174±2 | 7.41±.09 | 11.0 | 31.7±.4 |
| 8 | 5387 | 0.120 | | | | | | | |
| | | | 0.149 | $2.47x10^4$ | 0.188 | 114±2 | 2.82±.05 | 3.61 | 15.0±.3 |
| 9 | 7152 | 0.185 | | | | | | | |
| | | | 0.219 | $1.56x10^4$ | 0.148 | 35.4±.6 | .552±.009 | .785 | 3.73±.06 |
| 10 | 8642 | 0.260 | | | | | | | |
| | | | 0.306 | $1.10x10^4$ | 0.141 | 21.2±.3 | .233±.003 | .233 | 1.65±.02 |
| 11 | 9647 | 0.360 | | | | | | | |

For the sample data set of Table D5 this would be the concentrations in Table D7, column 10 plotted against the sizes in column 4. The upper error bar is the value plus the 90% confidence interval. The lower error bar is the value minus the 90% confidence interval. For $\bar{\alpha}_{3-4}$ in Table D7 we would have $\bar{\alpha}_{3-4}$ = 33.2 ± 0.6

thus:

$$\Delta N_S/\Delta \mathrm{LogD} = \frac{33.2 \times 4.76 \times 10}{0.250} \quad \frac{0.6 \times 4.76 \times 10}{0.250}$$

$$= (63.2 \pm 1.1) \times 10^6$$

The data shown in column 10, Table D7 is graphically displayed in Figure D11.

## D.4 OPTICAL PARTICLE COUNTERS

Optical particle counters are a useful adjunct to the ultrafine sizing system, giving additional data in the region where impactors, diffusional and electrical methods have the worst resolution.

A number of commercial optical particle counters are available which will perform adequately in the field environment associated with precipitator evaluation. These instruments can be obtained with several optical sensor configurations and may usually be specified to perform in a threshold mode, where all particles larger than a certain size are counted, or in a window mode where a narrow range of particle sizes is indicated. In either instance, the smallest particle which will be detected is approximately 0.3 μm diameter. In this respect, most optical counters size particles in the same range as cascade impactors and cyclones.

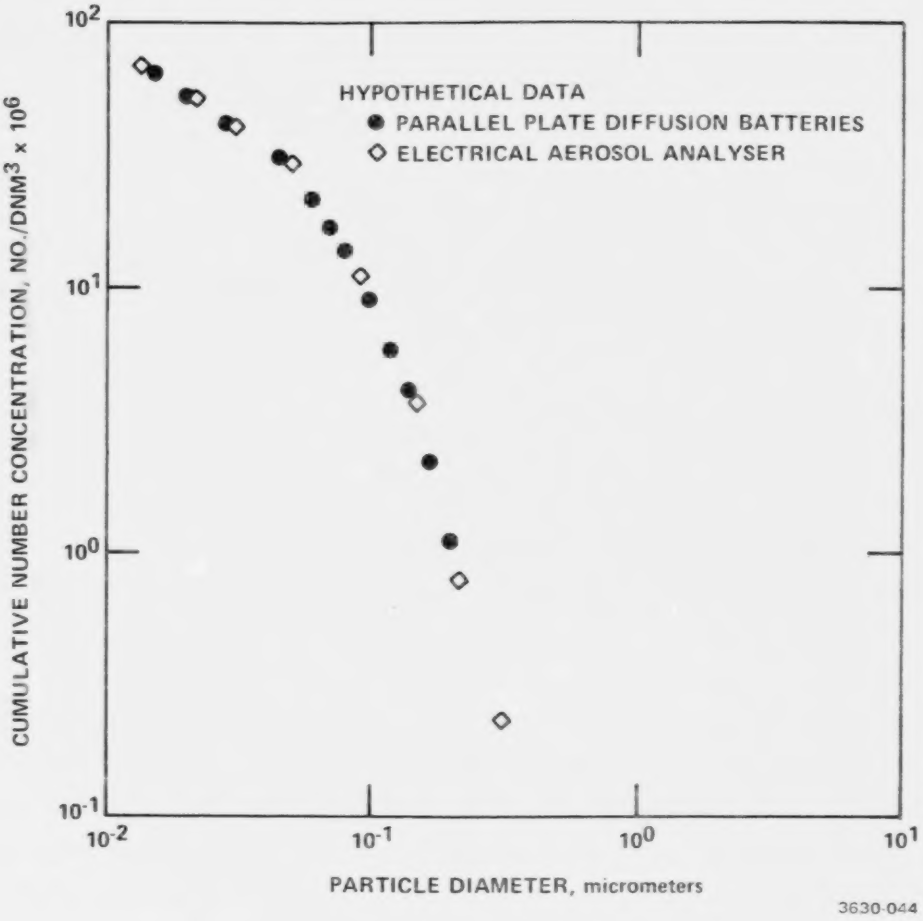


*Figure D11. Hypothetical inlet size distribution at a fabric filter on a coal-fired boiler. Sample data for parallel plate diffusion batteries and electrical aerosol analyser are shown.*

The calibration of optical particle counters is usually done with monodisperse plastic spheres which have a refractive index of 1.59. Any deviations from this index of refraction, or sphericity of the particles will cause the indicated sizes to be in error. If high accuracy in the optical particle counter data is important to the test, it is possible to do calibrations in the field, using the test aerosol. McCain (1974) has used a sedimentation technique which employs parallel plate diffusion batteries as sedimentation chambers to correlate aerodynamic particle diameter with the indicated, or equivalent PSL, diameter. Marple and Rubow (1976) reported the development of special impaction devices which may be used for this same purpose.

In the majority of full sampling situations, optical particle counters must be used with some type of Sample Extraction and Dilution System as described in Section D.1. The sample is thus cooled and diluted to less than 300 particles per $cm^3$. Also it is usually necessary to place the return line back into the dilution system to minimize pressure gradients across the counter system.

Most commercially available systems come with real-time analog outputs and digital outputs with one to 10 minute accumulation or integration times. Analog signals are particularly useful for monitoring source variations and rapping emissions.

Data accumulated by optical particle counters are given as particles per unit volume and, after multiplication by the proper dilution factor, may be plotted on a cumulative or differential number basis. See paragraph C.8 for a discussion of data plotting. Figure 34 in Section 2.3.4. shows data taken using an optical-diffusional impactor system.

# APPENDIX E

## SUMMARY OF SOURCE PERFORMANCE REFERENCE METHODS

To evaluate the performance standards for new stationary sources, the EPA has promulgated reference methods which specify the manner in which certain tests must be performed. These Reference Methods can be found in the Code of Federal Regulations under Title 40 - Protection of Environment; Chapter 1 - Environmental Protection Agency; Subchapter C - Air Programs; Part 60 - Standards of Performance for New Stationary Sources; Appendix A - Reference Methods.

In the first section of this appendix these reference methods are summarized. In the latter part of this appendix these reference methods, as presented in the Code of Federal Regulations, are reproduced verbatim. They have been corrected and amended through April 1, 1978.

## Title 40—Protection of Environment

## CHAPTER I—ENVIRONMENTAL PROTECTION AGENCY

[FRL 754-5]

## PART 60—STANDARDS OF PERFORMANCE FOR NEW STATIONARY SOURCES

### Revision to Reference Methods 1–8

AGENCY: Environmental Protection Agency.

ACTION: Final Rule.

SUMMARY: This rule revises Reference Methods 1 through 8, the detailed requirements used to measure emissions from affected facilities to determine whether they are in compliance with a standard of performance. The methods were originally promulgated December 23, 1971, and since that time several revisions became apparent which would clarify, correct and improve the methods. These revisions make the methods easier to use, and improve their accuracy and reliability.

EFFECTIVE DATE: September 19, 1977.

ADDRESSES: Copies of the comment letters are available for public inspection and copying at the U.S. Environmental Protection Agency, Public Information Reference Unit (EPA Library), Room 2922, 401 M Street, S.W., Washington, D.C. 20460. A summary of the comments and EPA's responses may be obtained upon written request from the EPA Public Information Center (PM-215), 401 M Street, S.W., Washington, D.C. 20460 (specify "Public Comment Summary: Revisions to Reference Methods 1–8 in Appendix A of Standards of Performance for New Stationary Sources").

FOR FURTHER INFORMATION CONTACT:

Don R. Goodwin, Emission Standards and Engineering Division, Environmental Protection Agency, Research Triangle Park, North Carolina 27711, telephone No. 919–541–5271.

SUPPLEMENTARY INFORMATION: The amendments were proposed on June 8, 1976 (40 FR 23060). A total of 55 comment letters were received during the comment period—34 from industry, 15 from governmental agencies, and 6 from other interested parties. They contained numerous suggestions which were incorporated in the final revisions.

Changes common to all eight of the reference methods are: (1) the clarification of procedures and equipment specifications resulting from the comments, (2) the addition of guidelines for alternative procedures and equipment to make prior approval of the Administrator unnecessary and (3) the addition of an introduction to each reference method discussing the general use of the method and delineating the procedure for using alternative methods and equipment.

Specific changes to the methods are:

### METHOD 1

1. The provision for the use of more than two traverse diameters, when specified by the Administrator, has been deleted. If one traverse diameter is in a plane containing the greatest expected concentration variation, the intended purpose of the deleted paragraph will be fulfilled.

2. Based on recent data from Fluidyne (Particulate Sampling Strategies for Large Power Plants Including Nonuniform Flow, EPA-600/2-76-170, June 1976) and Entropy Environmentalists (Determination of the Optimum Number of Traverse Points: An Analysis of Method 1 Criteria (draft), Contract No. 68-01-3172), the number of traverse points for velocity measurements has been reduced and the 2:1 length to width ratio requirement for cross-sectional layout of rectangular ducts has been replaced by a "balanced matrix" scheme.

3. Guidelines for sampling in stacks containing cyclonic flow and stacks smaller than about 0.31 meter in diameter or 0.071 $m^2$ in cross-sectional area will be published at a later date.

4. Clarification has been made as to when a check for cyclonic flow is necessary; also, the suggested procedure for determination of unacceptable flow conditions has been revised.

### METHOD 2

1. The calibration of certain pitot tubes has been made optional. Appropriate construction and application guidelines have been included.

2. A detailed calibration procedure for temperature gauges has been included.

3. A leak check procedure for pitot lines has been included.

### METHOD 3

1. The applicablility of the method has

been confined to fossil-fuel combustion processes and to other processes where it has been determined that components other than $O_2$, $CO_2$, CO, and $N_2$ are not present in concentrations sufficient to affect the final results.

2. Based on recent research information (Particulate Sampling Strategies for Large Power Plants Including Nonuniform Flow, EPA-600/2-76-170, June 1976), the requirement for proportional sampling has been dropped and replaced with the requirement for constant rate sampling. Proportional and constant rate sampling have been found to give essentially the same result.

3. The "three consecutive" requirement has been replaced by "any three" for the determination of molecular weight, $CO_2$ and $O_2$.

4. The equation for excess air has been revised to account for the presence of CO.

5. A clearer distinction has been made between molecular weight determination and emission rate correction factor determination.

6. Single point, integrated sampling has been included.

### METHOD 4

1. The sampling time of 1 hour has been changed to a total sampling time which will span the length of time the pollutant emission rate is being determined or such time as specified in an applicable subpart of the standards.

2. The requirement for proportional sampling has been dropped and replaced with the requirement for constant rate sampling.

3. The leak check before the test run has been made optional; the leak check after the run remains mandatory.

### METHOD 5

1. The following alternatives have been included in the method:

a. The use of metal probe liners.

b. The use of other materials of construction for filter holders and probe liner parts.

c. The use of polyethylene wash bottles and sample storage containers.

d. The use of desiccants other than silica gel or calcium sulfate, when appropriate.

e. The use of stopcock grease other than silicone grease, when appropriate.

f. The drying of filters and probe-filter catches at elevated temperatures, when appropriate.

g. The combining of the filter and probe washes into one container.

2. The leak check prior to a test run has been made optional. The post-test leak check remains mandatory. A method for correcting sample volume for excessive leakage rates has been included.

3. Detailed leak check and calibration procedures for the metering system have been included.

### METHOD 6

1. Possible interfering agents of the method have been delineated.

2. The options of: (a) using a Method 8 impinger system, or (b) determining $SO_2$ simultaneously with particulate matter, have been included in the method.

3. Based on recent research data, the requirement for proportional sampling has been dropped and replaced with the requirement for constant rate sampling.

4. Tests have shown that isopropanol obtained from commercial sources occasionally has peroxide impurities that will cause erroneously low $SO_2$ measurements. Therefore, a test for detecting peroxides in isopropanol has been included in the method.

5. The leak check before the test run has been made optional; the leak check after the run remains mandatory.

6. A detailed calibration procedure for the metering system has been included in the method.

### METHOD 7

1. For variable wave length spectrophotometers, a scanning procedure for determining the point of maximum absorbance has been incorporated as an option.

### METHOD 8

1. Known interfering compounds have been listed to avoid misapplication of the method.

2. The determination of filterable particulate matter (including acid mist) simultaneously with $SO_3$ and $SO_2$ has been allowed where applicable.

3. Since occassionally some commercially available quantities of isopropanol have peroxide impurities that will cause erroneously high sulfuric acid mist measurements, a test for peroxides in isopropanol has been included in the method.

4. The gravimetric technique for moisture content (rather than volumetric) has been specified because a mixture of isopropyl alcohol and water will have a volume less than the sum of the volumes of its content.

5. A closer correspondence has been made between similar parts of Methods 8 and 5.

### MISCELLANEOUS

Several commenters questioned the meaning of the term "subject to the approval of the Administrator" in relation to using alternate test methods and procedures. As defined in § 60.2 of subpart A, the "Administrator" includes any authorized representative of the Administrator of the Environmental Protection Agency. Authorized representatives are EPA officials in EPA Regional Offices or State, local, and regional governmental officials who have been delegated the responsibility of enforcing regulations under 40 CFR 60. These officials in consultation with other staff members familiar with technical aspects of source testing will render decisions regarding acceptable alternate test procedures.

In accordance with section 117 of the Act, publication of these methods was preceded by consultation with appropriate advisory committees, independent experts, and Federal departments and agencies.

(Secs. 111, 114 and 301(a) of the Clean Air Act, sec. 4(a) of Pub. L. No. 91-604, 84 Stat

**1683; sec. 4(a) of Pub. L. No. 91–604, 84 Stat 1687; sec. 2 of Pub. L. No. 90–148, 81 Stat. 504 [42 U.S.C. 1857c–6, 1857c–9, 1857g(a)].)**

**NOTE.—The Environmental Protection Agency has determined that this document does not contain a major proposal requiring preparation of an Economic Impact Analysis under Executive Orders 11821 and 11949 and OMB Circular A–107.**

Dated: August 10, 1977.

DOUGLAS M. COSTLE,
*Administrator.*

**Part 60 of Chapter I of Title 40 of the Code of Federal Regulations is amended by revising Methods 1 through 8 of Appendix A—Reference Methods as follows:**

## APPENDIX A—REFERENCE METHODS

The reference methods in this appendix are referred to in § 60.8 (Performance Tests) and § 60.11 (Compliance With Standards and Maintenance Requirements) of 40 CFR Part 60, Subpart A (General Provisions). Specific uses of these reference methods are described in the standards of performance contained in the subparts, beginning with Subpart D.

Within each standard of performance, a section titled "Test Methods and Procedures" is provided to (1) identify the test methods applicable to the facility subject to the respective standard and (2) identify any special instructions or conditions to be followed when applying a method to the respective facility. Such instructions (for example, establish sampling rates, volumes, or temperatures) are to be used either in addition to, or as a substitute for procedures in a reference method. Similarly, for sources subject to emission monitoring requirements, specific instructions pertaining to any use of a reference method are provided in the subpart or in Appendix B.

Inclusion of methods in this appendix is not intended as an endorsement or denial of their applicability to sources that are not subject to standards of performance. The methods are potentially applicable to other sources; however, applicability should be confirmed by careful and appropriate evaluation of the conditions prevalent at such sources.

The approach followed in the formulation of the reference methods involves specifications for equipment, procedures, and performance. In concept, a performance specification approach would be preferable in all methods because this allows the greatest flexibility to the user. In practice, however, this approach is impractical in most cases because performance specifications cannot be established. Most of the methods described herein, therefore, involve specific equipment specifications and procedures, and only a few methods in this appendix rely on performance criteria.

Minor changes in the reference methods should not necessarily affect the validity of the results and it is recognized that alternative and equivalent methods exist. Section 60.8 provides authority for the Administrator to specify or approve (1) equivalent methods, (2) alternative methods, and (3) minor changes in the methodology of the reference methods. It should be clearly understood that unless otherwise identified all such methods and changes must have prior approval of the Administrator. An owner employing such methods or deviations from the reference methods without obtaining prior approval does so at the risk of subsequent disapproval and retesting with approved methods.

Within the reference methods, certain specific equipment or procedures are recognized as being acceptable or potentially acceptable and are specifically identified in the methods. The items identified as acceptable options may be used without approval but must be identified in the test report. The potentially approvable options are cited as "subject to the approval of the Administrator" or as "or equivalent." Such potentially approvable techniques or alternatives may be used at the discretion of the owner without prior approval. However, detailed descriptions for applying these potentially approvable techniques or alternatives are not provided in the reference methods. Also, the potentially approvable options are not necessarily acceptable in all applications. Therefore, an owner electing to use such potentially approvable techniques or alternatives is responsible for: (1) assuring that the techniques or alternatives are in fact applicable and are properly executed; (2) including a written description of the alternative method in the test report (the written method must be clear and must be capable of being performed without additional instruction, and the degree of detail should be similar to the detail contained in the reference methods); and (3) providing any rationale or supporting data necessary to show the validity of the alternative in the particular application. Failure to meet these requirements can result in the Administrator's disapproval of the alternative.

## METHOD 1

### Sample and Velocity Traverses for Stationary Sources:

Procedure for selecting stack sampling site and selecting and locating minimum number of traverse points.

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Regulation Promulgated | 36 | 247 | 12/23/71 | 24882 |
| Regulation Amended | 38 | 99 | 5/23/73 | 13562 |
| Proposed Revised Regulation | 41 | 111 | 6/08/76 | 23061 |
| Revised Regulation Promulgated | 42 | 160 | 8/18/77 | 41755 |

## METHOD 2

### Determination of Stack Gas and Volumetric Flowrate:

Procedure for determining stack gas velocity from gas density and velocity head using Type S pitot tube. Volumetric flowrate is calculated from gas velocity and stack cross-sectional area.

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Regulation Promulgated | 36 | 247 | 12/23/71 | 24386 |
| Proposed Revised Regulation | 41 | 111 | 6/08/76 | 23063 |
| Revised Regulation Promulgated | 42 | 160 | 8/18/77 | 41758 |

## METHOD 3

### Gas Analysis for Carbon Dioxide, Excess Air, and Dry Molecular Weight:

$CO_2$, CO, and $O_2$ are determined by absorbing in appropriate reagents (Orsat analysis).

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Regulation Promulgated | 36 | 247 | 12/23/71 | 24886 |
| Proposed Revised Regulation | 41 | 111 | 6/08/76 | 23069 |
| Revised Regulation Promulgated | 42 | 160 | 8/18/77 | 41768 |

## METHOD 4

### Determination of Moisture in Stack Gases:

Stack gas moisture content is determined by condensation and measuring condensed water volumetrically.

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Regulation Promulgated | 36 | 247 | 12/23/71 | 24887 |
| Proposed Revised Regulation | 41 | 111 | 6/08/76 | 23072 |
| Revised Regulation Promulgated | 42 | 160 | 8/18/77 | 41771 |

## METHOD 5

### Determination of Particulate Emissions from Stationary Sources:

Stack gas is sampled isokinetically, particulate matter filtered and weighed after removal of uncombined water.

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Regulation Promulgated | 36 | 247 | 12/23/71 | 24888 |
| Regulation Amended | 38 | 99 | 5/23/73 | 13563 |
| Proposed Revised Regulation | 41 | 111 | 6/08/76 | 23076 |
| Revised Regulation Promulgated | 42 | 160 | 8/18/77 | 41776 |

## METHOD 6

## Determination of Sulfur Dioxide Emissions from Stationary Sources:

$SO_2$ is separated from any $SO_3$ and $H_2SO_4$ mist present using aqueous isopropyl alcohol. The $SO_2$ is passed through dilute hydrogen peroxide which is titrated by the barium-thoris method to determine sulfate formation.

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Regulation Promulgated | 36 | 247 | 12/23/71 | 24890 |
| Proposed Revised Regulation | 41 | 111 | 6/08/76 | 23083 |
| Revised Regulation Promulgated | 42 | 160 | 8/18/77 | 41782 |

## METHOD 7

## Determination of Nitrogen Oxide Emissions from Stationary Sources:

A grab sample is drawn into an evacuated flask containing actified hydrogen peroxide which converts NO and $NO_2$ to $HNO_3$. Analysis is made by the colorimetric PDS method.

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Regulation Promulgated | 36 | 247 | 12/23/71 | 24891 |
| Proposed Revised Regulation | 41 | 111 | 6/08/76 | 23085 |
| Revised Regulation Promulgated | 42 | 160 | 8/18/77 | 41784 |

## METHOD 8

### Determination of Sulfuric Acid Mist and Sulfur Dioxide Emissions from Stationary Sources:

$SO_3$ and $H_2SO_4$ mist are absorbed in aqueous isopropyl alcohol and $SO_2$ passes through to be collected in hydrogen peroxide solution. Each solution is titrated by the barium-thoris method to determine sulfate formation.

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Regulation Promulgated | 36 | 247 | 12/23/71 | 24893 |
| Proposed Revised Regulation | 41 | 111 | 6/08/76 | 23087 |
| Revised Regulation Promulgated | 42 | 160 | 8/18/77 | 41786 |

## METHOD 9

### Visual Determination of the Opacity of Emissions from Stationary Sources:

Opacity of emissions from a stationary source is determined visually by qualified observers.

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Regulation Promulgated | 36 | 247 | 12/23/71 | 24895 |
| Proposed Revised Regulation | 39 | 177 | 9/11/74 | 32857 |
| Revised Regulation Promulgated | 39 | 219 | 11/12/74 | 39874 |

## METHOD 10

### Determination of Carbon Monoxide Emissions from Stationary Sources:

Integrated or continuous gas samples from stacks are analysed for CO content using NDIR analysis.

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Regulation Promulgated | 39 | 47 | 3/08/74 | 9319 |
| Regulation Amended | 39 | 75 | 4/07/74 | 13776 |

## METHOD 11

### Determination of Hydrogen Sulfide Emissions from Stationary Sources:

$H_2S$ is collected in alkaline $Cd(OH)_2$ to form CdS, which is dissolved in HCL and reacted with a known amount of iodine. Amount of iodine consumed is measure of $H_2S$ present in gas.

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Regulation Promulgated | 39 | 47 | 3/08/74 | 9321 |
| Regulation Amended | 39 | 75 | 4/17/74 | 13776 |
| Proposed Revised Regulation | 42 | 99 | 5/23/77 | 26222 |

## METHOD 12

### Determination of Sulfur Dioxide Emissions from Stationary Sources by Continuous Monitors:

No specific continuous monitor required; results must show acceptable relationship to those determined by Method 6 or 8.

(RESERVED)

## METHOD 13A

### Determination of Total Fluoride Emissions from Stationary Sources - SPADNS Zirconium Lake Method:

Gaseous and particulate fluorides are withdrawn isokinetically and concentration determined by SPADNS method.

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Regulation Promulgated | 40 | 152 | 8/06/76 | 33157 |
| Regulation Amended | 41 | 230 | 11/29/76 | 52299 |

## METHOD 13B

### Determination of Total Fluoride Emissions from Stationary Sources - Specific Ion Electrode Method:

Gaseous and particulate fluorides are withdrawn isokinetically and concentration determined by a specific ion electrode.

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Regulation Promulgated | 40 | 152 | 8/06/75 | 33163 |
| Regulation Amended | 41 | 230 | 11/29/76 | 52299 |

## METHOD 14

### Determination of Fluoride Emissions from Pot Room Roof Monitors of Primary Aluminum Plants:

A permanent sampling manifold is constructed to isokinetically withdraw sample gas from the roof monitor. The gas sample is brought to ground level and sampled using Method 13A or 13B for determination of fluoride concentration.

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Regulation Promulgated | 41 | 17 | 1/26/76 | 3829 |

## METHOD 15

### Determination of $H_2S$, COS, and $CS_2$ Emissions from Stationary Sources:

A gas sample is extracted and diluted with clean dry air. An aliquot of the diluted sample is then analysed by gas chromatographic separation (GC) and flame photometric detection (FPD).

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Proposed Regulation | 41 | 193 | 10/04/76 | 43870 |
| Regulation Promulgated | 43 | 51 | 3/15/78 | 10870 |

## METHOD 16

### Semicontinuous Determination of Sulfur Emissions from Stationary Sources:

A gas sample is extracted and diluted with clean dry air. An aliquot of the diluted sample is then analysed for gas sulfur. Two GC/FPD analysers are used for resolution of both high and low molecular weight sulfur compounds.

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Proposed Regulation | 41 | 187 | 9/24/76 | 42017 |
| Regulation Promultaged | 43 | 37 | 2/23/78 | 7575 |

## METHOD 17

### Determination of Particulate Emissions from Stationary Sources (In-Stack Filtration Method):

Particulate matter is withdrawn isokinetically and collected on a filter maintained at stack temperature. Particulate matter mass is determined gravimetrically after removal of uncombined $H_2O$.

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Proposed Regulation | 41 | 187 | 9/24/76 | 42020 |
| Regulation Promulgated | 43 | 37 | 2/23/78 | 7585 |

# METHOD 1

# SAMPLE AND VELOCITY TRAVERSES FOR STATIONARY SOURCES

## 1 *Principle and Applicability*

1.1 Principle. To aid in the representative measurement of pollutant emissions and/or total volumetric flow rate from a stationary source, a measurement site where the effluent stream is flowing in a known direction is selected, and the cross-section of the stack is divided into a number of equal areas. A traverse point is then located within each of these equal areas.

1.2 Applicability. This method is applicable to flowing gas streams in ducts, stacks, and flues. The method cannot be used when: (1) flow is cyclonic or swirling (see Section 2.4), (2) a stack is smaller than about 0.30 meter (12 in.) in diameter, or 0.071 m² (113 in.²) in cross-sectional area, or (3) the measurement site is less than two stack or duct diameters downstream or less than a half diameter upstream from a flow disturbance.

The requirements of this method must be considered before construction of a new facility from which emissions will be measured; failure to do so may require subsequent alterations to the stack or deviation from the standard procedure. Cases involving variants are subject to approval by the Administrator, U.S. Environmental Protection Agency.

## 2. *Procedure*

2.1 Selection of Measurement Site. Sampling or velocity measurement is performed at a site located at least eight stack or duct diameters downstream and two diameters upstream from any flow disturbance such as a bend, expansion, or contraction in the stack, or from a visible flame. If necessary, an alternative location may be selected, at a position at least two stack or duct diameters downstream and a half diameter upstream from any flow disturbance. For a rectangular cross section, an equivalent diameter ($D_e$) shall be calculated from the following equation, to determine the upstream and downstream distances:

$$D_e = \frac{2LW}{L+W}$$

where L = length and W = width.

2.2 Determining the Number of Traverse Points.

2.2.1 Particulate Traverses. When the eight- and two-diameter criterion can be met, the minimum number of traverse points shall be: (1) twelve, for circular or rectangular stacks with diameters (or equivalent diameters) greater than 0.61 meter (24 in.); (2) eight, for circular stacks with diameters between 0.30 and 0.61 meter (12–24 in.); (3) nine, for rectangular stacks with equivalent diameters between 0.30 and 0.61 meter (12–24 in.).

When the eight- and two-diameter criterion cannot be met, the minimum number of traverse points is determined from Figure 1–1. Before referring to the figure, however, determine the distances from the chosen measurement site to the nearest upstream and downstream disturbances, and divide each distance by the stack diameter or equivalent diameter, to determine the distance in terms of the number of duct diameters. Then, determine from Figure 1–1 the minimum number of traverse points that corresponds: (1) to the number of duct diameters upstream; and (2) to the number of diameters downstream. Select the higher of the two minimum numbers of traverse points, or a greater value, so that for circular stacks the number is a multiple of 4, and for rectangular stacks, the number is one of those shown in Table 1–1.

TABLE 1–1. *Cross-sectional layout for rectangular stacks*

| *Number of traverse points:* | *Matrix layout* |
|---|---|
| 9 | 3x3 |
| 12 | 4x3 |
| 16 | 4x4 |
| 20 | 5x4 |
| 25 | 5x5 |
| 30 | 6x5 |
| 36 | 6x6 |
| 42 | 7x6 |
| 49 | 7x7 |

2.2.2 Velocity (Non-Particulate) Traverses. When velocity or volumetric flow rate is to be determined (but not particulate matter), the same procedure as that for particulate traverses (Section 2.2.1) is followed, except that Figure 1–2 may be used instead of Figure 1–1.

2.3 Cross-Sectional Layout and Location of Traverse Points.

2.3.1 Circular Stacks. Locate the traverse points on two perpendicular diameters according to Table 1–2 and the example shown in Figure 1–3. Any equation (for examples, see Citations 2 and 3 in the Bibliography) that gives the same values as those in Table 1–2 may be used in lieu of Table 1–2.

For particulate traverses, one of the diameters must be in a plane containing the greatest expected concentration variation, e.g., after bends, one diameter shall be in the plane of the bend. This requirement becomes less critical as the distance from the disturbance increases; therefore, other diameter locations may be used, subject to approval of the Administrator.

In addition, for stacks having diameters greater than 0.61 m (24 in.) no traverse points shall be located within 2.5 centimeters (1.00 in.) of the stack walls; and for stack diameters equal to or less than 0.61 m (24 in.), no traverse points shall be located within 1.3 cm (0.50 in.) of the stack walls. To meet these criteria, observe the procedures given below.

2.3.1.1 Stacks With Diameters Greater Than 0.61 m (24 in.). When any of the traverse points as located in Section 2.3.1 fall within 2.5 cm (1.00 in.) of the stack walls, relocate them away from the stack walls to: (1) a distance of 2.5 cm (1.00 in.); or (2) a distance equal to the nozzle inside diameter, whichever is larger. These relocated traverse points (on each end of a diameter) shall be the "adjusted" traverse points.

Whenever two successive traverse points are combined to form a single adjusted traverse point, treat the adjusted point as two separate traverse points, both in the sampling (or velocity measurement) procedure, and in recording the data.

2.3.1.2 Stacks With Diameters Equal to or Less Than 0.61 m (24 in.). Follow the procedure in Section 2.3.1.1, noting only that any "adjusted" points should be relocated away from the stack walls to: (1) a distance of 1.3 cm (0.50 in.); or (2) a distance equal to the nozzle inside diameter, whichever is larger.

2.3.2 Rectangular Stacks. Determine the number of traverse points as explained in Sections 2.1 and 2.2 of this method. From Table 1-1, determine the grid configuration. Divide the stack cross-section into as many equal rectangular elemental areas as traverse points, and then locate a traverse point at the centroid of each equal area according to the example in Figure 1–4.

The situation of traverse points being too close to the stack walls is not expected to arise with rectangular stacks. If this problem should ever arise, the Administrator must be contacted for resolution of the matter.

2.4 Verification of Absence of Cyclonic Flow. In most stationary sources, the direction of stack gas flow is essentially parallel to the stack walls. However, cyclonic flow may exist (1) after such devices as cyclones and inertial demisters following venturi scrubbers, or (2) in stacks having tangential inlets or other duct configurations which tend to induce swirling; in these instances, the presence or absence of cyclonic flow at the sampling location must be determined. The following techniques are acceptable for this determination.

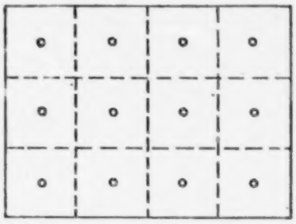


Figure 1-4. Example showing rectangular stack cross section divided into 12 equal areas, with a traverse point at centroid of each area.

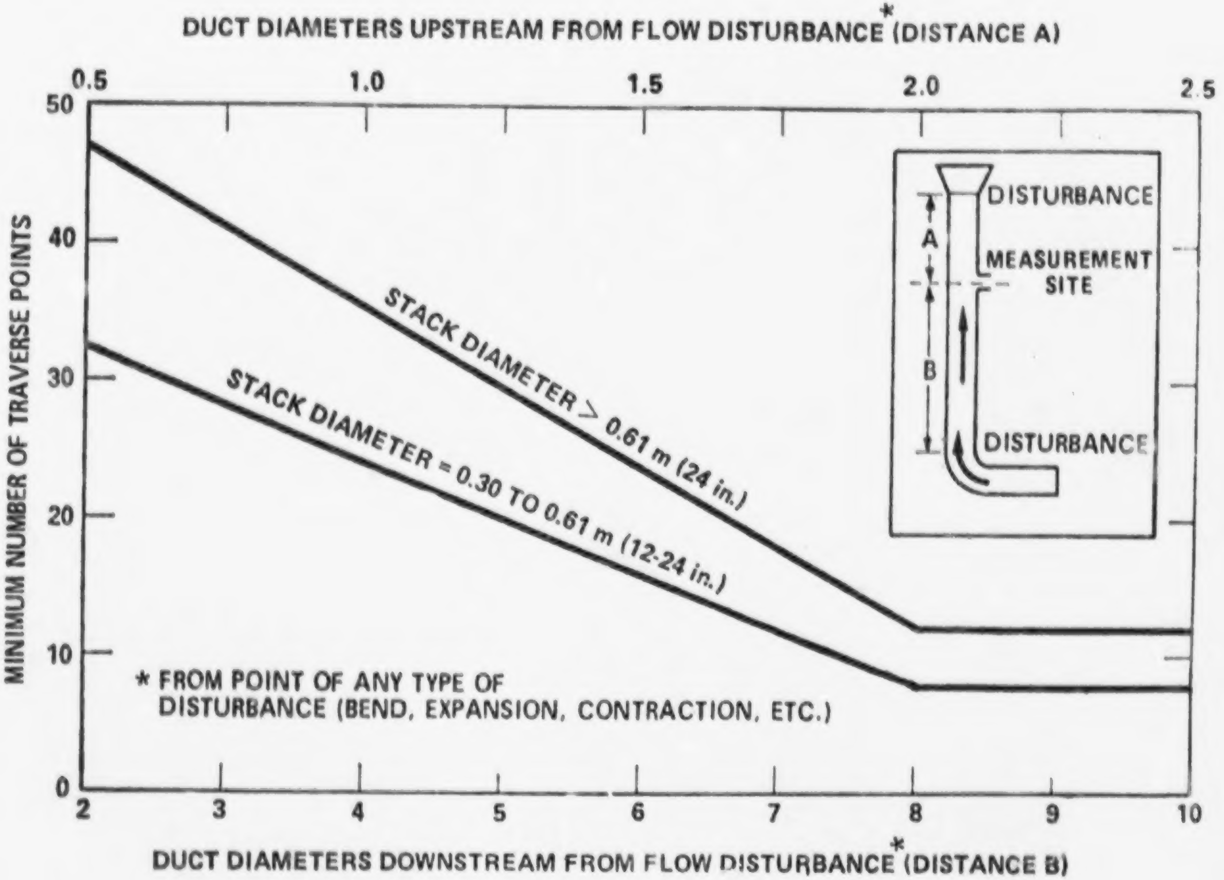


Figure 1-1. Minimum number of traverse points for particulate traverses.

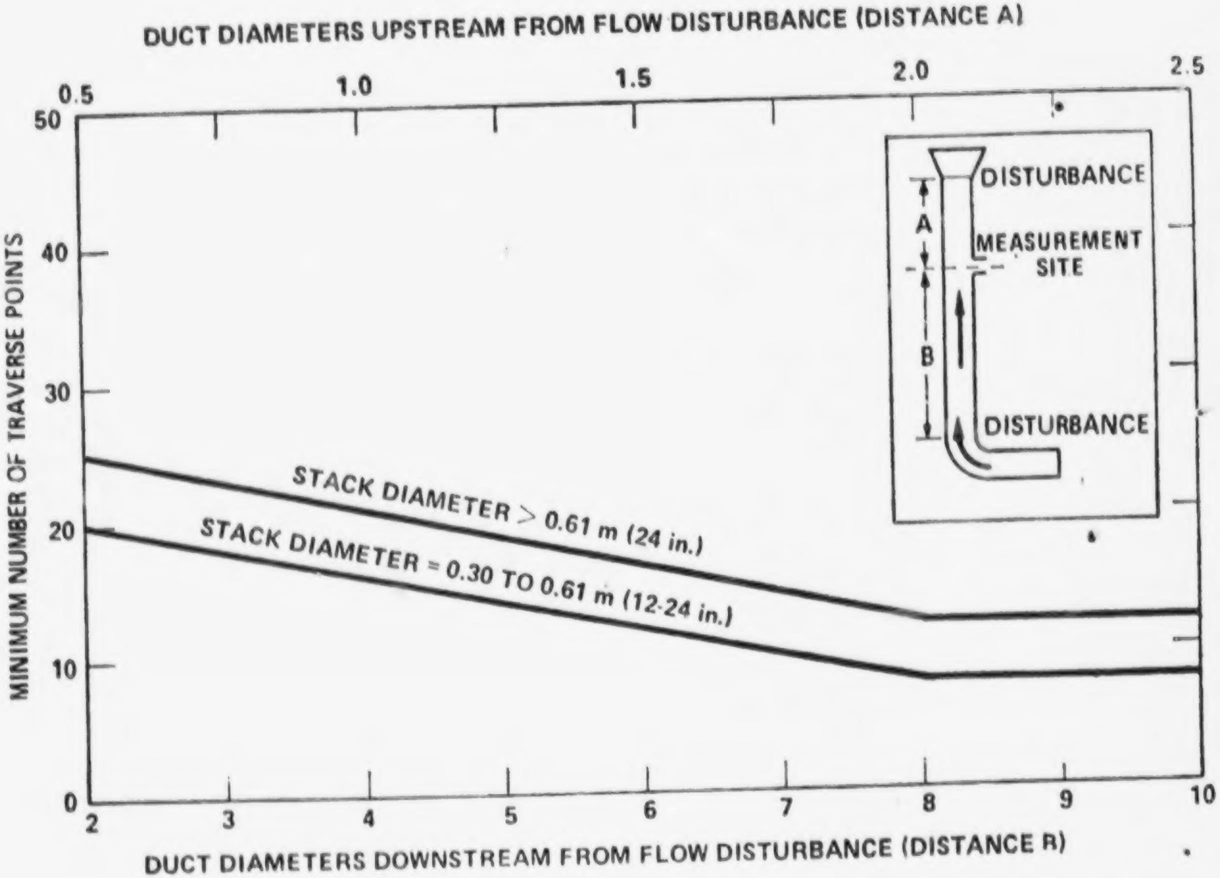


Figure 1-2. Minimum number of traverse points for velocity (nonparticulate) traverses.

Level and zero the manometer. Connect a Type S pitot tube to the manometer. Position the Type S pitot tube at each traverse point, in succession, so that the planes of the face openings of the pitot tube are perpendicular to the stack cross-sectional plane: when the Type S pitot tube is in this position, it is at "0° reference." Note the differential pressure (Δp) reading at each traverse point. If a null (zero) pitot reading is obtained at 0° reference at a given traverse point, an acceptable flow condition exists at that point. If the pitot reading is not zero at 0° reference, rotate the pitot tube (up to ±90° yaw angle), until a null reading is obtained. Carefully determine and record the value of the rotation angle ($\alpha$) to the nearest degree. After the null technique has been applied at each travrse point, calculate the average of the absolute values of $\alpha$; assign $\alpha$ values of 0° to those points for which no rotation was required, and include these in the overall average. If the average value of $\alpha$ is greater than 10°, the overall flow condition in the stack is unacceptable and alternative methodology, subject to the approval of the Administrator, must be used to perform accurate sample and velocity traverses.

### 3. *Bibliography*

1. Determining Dust Concentration in a Gas Stream. ASME. Performance Test Code No. 27. New York. 1957.

2. Devorkin, Howard, et al. Air Pollution Source Testing Manual. Air Pollution Control District. Los Angeles, CA. November 1963

3. Methods for Determination of Velocity, Volume, Dust and Mist Content of Gases. Western Precipitation Division of Joy Manufacturing Co. Los Angeles, CA. Bulletin WP-50. 1968.

4. Standard Method for Sampling Stacks for Particulate Matter. In: 1971 Book of ASTM Standards, Part 23. ASTM Designation D-2928-71. Philadelphia, Pa. 1971.

5. Hanson, H. A., et al. Particulate Sampling Strategies for Large Power Plants Including Nonuniform Flow. USEPA, ORD, ESRL, Research Triangle Park, N.C. EPA-600/2-76-170. June 1976.

6. Entropy Environmentalists, Inc. Determination of the Optimum Number of Sampling Points: An Analysis of Method 1 Criteria. Environmental Protection Agency. Research Triangle Park, N.C. EPA Contract No. 68-02-3172, Task 7.

| TRAVERSE POINT | DISTANCE, % of diameter |
|---|---|
| 1 | 4.4 |
| 2 | 14.7 |
| 3 | 29.5 |
| 4 | 70.5 |
| 5 | 85.3 |
| 6 | 95.6 |

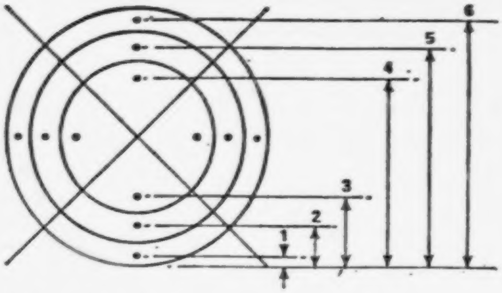


Figure 1-3. Example showing circular stack cross section divided into 12 equal areas, with location of traverse points indicated.

## Table 1-2. LOCATION OF TRAVERSE POINTS IN CIRCULAR STACKS

(Percent of stack diameter from inside wall to traverse point)

| Traverse point number on a diameter | Number of traverse points on a diameter | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 2 | 4 | 6 | 8 | 10 | 12 | 14 | 16 | 18 | 20 | 22 | 24 |
| 1 | 14.6 | 6.7 | 4.4 | 3.2 | 2.6 | 2.1 | 1.8 | 1.6 | 1.4 | 1.3 | 1.1 | 1.1 |
| 2 | 85.4 | 25.0 | 14.6 | 10.5 | 8.2 | 6.7 | 5.7 | 4.9 | 4.4 | 3.9 | 3.5 | 3.2 |
| 3 | | 75.0 | 29.6 | 19.4 | 14.6 | 11.8 | 9.9 | 8.5 | 7.5 | 6.7 | 6.0 | 5.5 |
| 4 | | 93.3 | 70.4 | 32.3 | 22.6 | 17.7 | 14.6 | 12.5 | 10.9 | 9.7 | 8.7 | 7.9 |
| 5 | | | 85.4 | 67.7 | 34.2 | 25.0 | 20.1 | 16.9 | 14.6 | 12.9 | 11.6 | 10.5 |
| 6 | | | 95.6 | 80.6 | 65.8 | 35.6 | 26.9 | 22.0 | 18.8 | 16.5 | 14.6 | 13.2 |
| 7 | | | | 89.5 | 77.4 | 64.4 | 36.6 | 28.3 | 23.6 | 20.4 | 18.0 | 16.1 |
| 8 | | | | 96.8 | 85.4 | 75.0 | 63.4 | 37.5 | 29.6 | 25.0 | 21.8 | 19.4 |
| 9 | | | | | 91.8 | 82.3 | 73.1 | 62.5 | 38.2 | 30.6 | 26.2 | 23.0 |
| 10 | | | | | 97.4 | 88.2 | 79.9 | 71.7 | 61.8 | 38.8 | 31.5 | 27.2 |
| 11 | | | | | | 93.3 | 85.4 | 78.0 | 70.4 | 61.2 | 39.3 | 32.3 |
| 12 | | | | | | 97.9 | 90.1 | 83.1 | 76.4 | 69.4 | 60.7 | 39.8 |
| 13 | | | | | | | 94.3 | 87.5 | 81.2 | 75.0 | 68.5 | 60.2 |
| 14 | | | | | | | 98.2 | 91.5 | 85.4 | 79.6 | 73.8 | 67.7 |
| 15 | | | | | | | | 95.1 | 89.1 | 83.5 | 78.2 | 72.8 |
| 16 | | | | | | | | 98.4 | 92.5 | 87.1 | 82.0 | 77.0 |
| 17 | | | | | | | | | 95.6 | 90.3 | 85.4 | 80.6 |
| 18 | | | | | | | | | 98.6 | 93.3 | 88.4 | 83.9 |
| 19 | | | | | | | | | | 96.1 | 91.3 | 86.8 |
| 20 | | | | | | | | | | 98.7 | 94.0 | 89.5 |
| 21 | | | | | | | | | | | 96.5 | 92.1 |
| 22 | | | | | | | | | | | 98.9 | 94.5 |
| 23 | | | | | | | | | | | | 96.8 |
| 24 | | | | | | | | | | | | 98.9 |

# METHOD 2

# DETERMINATION OF STACK GAS AND VOLUMETRIC FLOWRATE

## 1. *Principle and Applicability*

1.1 Principle. The average gas velocity in a stack is determined from the gas density and from measurement of the average velocity head with a Type S (Stausscheibe or reverse type) pitot tube.

1.2 Applicability. This method is applicable for measurement of the average velocity of a gas stream and for quantifying gas flow.

This procedure is not applicable at measurement sites which fail to meet the criteria of Method 1, Section 2.1. Also, the method cannot be used for direct measurement in cyclonic or swirling gas streams; Section 2.4 of Method 1 shows how to determine cyclonic or swirling flow conditions. When unacceptable conditions exist, alternative procedures, subject to the approval of the Administrator, U.S. Environmental Protection Agency, must be employed to make accurate flow rate determinations; examples of such alternative procedures are: (1) to install straightening vanes; (2) to calculate the total volumetric flow rate stoichiometrically, or (3) to move to another measurement site at which the flow is acceptable.

## 2. *Apparatus*

Specifications for the apparatus are given below. Any other apparatus that has been demonstrated (subject to approval of the Administrator) to be capable of meeting the specifications will be considered acceptable.

2.1 Type S Pitot Tube. The Type S pitot tube (Figure 2-1) shall be made of metal tubing (e.g., stainless steel). It is recommended that the external tubing diameter (dimension $D_t$, Figure 2-2b) be between 0.48 and 0.95 centimeters ($\frac{3}{16}$ and $\frac{3}{8}$ inch). There shall be an equal distance from the base of each leg of the pitot tube to its face-opening plane (dimensions $P_A$ and $P_B$, Figure 2-2b); it is recommended that this distance be between 1.05 and 1.50 times the external tubing diameter. The face openings of the pitot tube shall, preferably, be aligned as shown in Figure 2-2; however, slight misalignments of the openings are permissible (see Figure 2-3).

The Type S pitot tube shall have a known coefficient, determined as outlined in Section 4. An identification number shall be assigned to the pitot tube; this number shall be permanently marked or engraved on the body of the tube.

A standard pitot tube may be used instead of a Type S, provided that it meets the specifications of Sections 2.7 and 4.2; note, however, that the static and impact pressure holes of standard pitot tubes are susceptible to plugging in particulate-laden gas streams. Therefore, whenever a standard pitot tube is used to perform a traverse, adequate proof must be furnished that the openings of the pitot tube have not plugged up during the traverse period; this can be done by taking a velocity head ($\Delta p$) reading at the final traverse point, cleaning out the impact and static holes of the standard pitot tube by "back-purging" with pressurized air, and then taking another $\Delta p$ reading. If the $\Delta p$ readings made before and after the air purge are the same ($\pm 5$ percent), the traverse is acceptable. Otherwise, reject the run. Note that if $\Delta p$ at the final traverse point is unsuitably low, another point may be selected. If "back-purging" at regular intervals is part of the procedure, then comparative $\Delta p$ readings shall be taken, as above, for the last two back purges at which suitably high $\Delta p$ readings are observed.

2.2 Differential Pressure Gauge. An inclined manometer or equivalent device is used. Most sampling trains are equipped with a 10-in. (water column) inclined-vertical manometer, having 0.01-in. $H_2O$ divisions on the 0- to 1-in. inclined scale, and 0.1-in. $H_2O$ divisions on the 1- to 10-in. vertical scale. This type of manometer (or other gauge of equivalent sensitivity) is satisfactory for the measurement of $\Delta p$ values as low as 1.3 mm (0.05 in.) $H_2O$. However, a differential pressure gauge of greater sensitivity shall be used (subject to the approval of the Administrator), if any of the following is found to be true: (1) the arithmetic average of all $\Delta p$ readings at the traverse points in the stack is less than 1.3 mm (0.05 in.) $H_2O$; (2) for traverses of 12 or more points, more than 10 percent of the individual $\Delta p$ readings are below 1.3 mm (0.05 in.) $H_2O$; (3) for traverses of fewer than 12 points, more than one $\Delta p$ reading is below 1.3 mm (0.05 in.) $H_2O$. Citation 18 in Section 6 describes commercially available instrumentation for the measurement of low-range gas velocities.

As an alternative to criteria (1) through (3) above, the following calculation may be performed to determine the necessity of using a more sensitive differential pressure gauge:

$$T=\frac{\sum_{i=1}^{n}\sqrt{\Delta p_i+K}}{\sum_{i=1}^{n}\sqrt{\Delta p_i}}$$

where:

$\Delta p_i$ = Individual velocity head reading at a traverse point, mm $H_2O$ (in. $H_2O$).

$n$ = Total number of traverse points.

$K$ = 0.13 mm $H_2O$ when metric units are used and 0.005 in $H_2O$ when English units are used.

If $T$ is greater than 1.05, the velocity head data are unacceptable and a more sensitive differential pressure gauge must be used.

NOTE.—If differential pressure gauges other than inclined manometers are used (e.g., magnehelic gauges), their calibration must be checked after each test series. To check the calibration of a differential pressure gauge, compare $\Delta p$ readings of the gauge with those of a gauge-oil manometer at a minimum of three points, approximately representing the range of $\Delta p$ values in the stack. If, at each point, the values of $\Delta p$ as read by the differential pressure gauge and gauge-oil manometer agree to within 5 percent, the differential pressure gauge shall be considered to be in proper calibration. Otherwise, the test series shall either be voided, or procedures to adjust the measured $\Delta p$ values and final results shall be used, subject to the approval of the Administrator.

2.3 Temperature Gauge. A thermocouple, liquid-filled bulb thermometer, bimetallic thermometer, mercury-in-glass thermometer, or other gauge capable of measuring temperature to within 1.5 percent of the minimum absolute stack temperature shall be used. The temperature gauge shall be attached to the pitot tube such that the sensor tip does not touch any metal; the gauge shall be in an interference-free arrangement with respect to the pitot tube face openings (see Figure 2-1 and also Figure 2-7 in Section 4). Alternate positions may be used if the pitot tube-temperature gauge system is calibrated according to the procedure of Section 4. Provided that a difference of not more than 1 percent in the average velocity measurement is introduced, the temperature gauge need not be attached to the pitot tube; this alternative is subject to the approval of the Administrator.

2.4 Pressure Probe and Gauge. A piezometer tube and mercury- or water-filled U-tube manometer capable of measuring stack pressure to within 2.5 mm (0.1 in.) Hg is used. The static tap of a standard type pitot tube or one leg of a Type S pitot tube with the face opening planes positioned parallel to the gas flow may also be used as the pressure probe.

2.5 Barometer. A mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg) may be used. In many cases, the barometric reading may be obtained from a nearby national weather service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and the sampling point shall be applied at a rate of minus 2.5 mm (0.1 in.) Hg per 30-meter (100 foot) elevation increase, or vice-versa for elevation decrease.

2.6 Gas Density Determination Equipment. Method 3 equipment, if needed (see Section 3.6), to determine the stack gas dry molecular weight, and Reference Method 4 or Method 5 equipment for moisture content determination; other methods may be used subject to approval of the Administrator.

2.7 Calibration Pitot Tube. When calibration of the Type S pitot tube is necessary (see Section 4), a standard pitot tube is used as a reference. The standard pitot tube shall, preferably, have a known coefficient, obtained either (1) directly from the National Bureau of Standards, Route 270, Quince Orchard Road, Gaithersburg, Maryland, or (2) by calibration against another standard pitot tube with an NBS-traceable coefficient. Alternatively, a standard pitot tube designed according to the criteria given in 2.7.1 through 2.7.5 below and illustrated in Figure 2-4 (see also Citations 7, 9, and 17 in

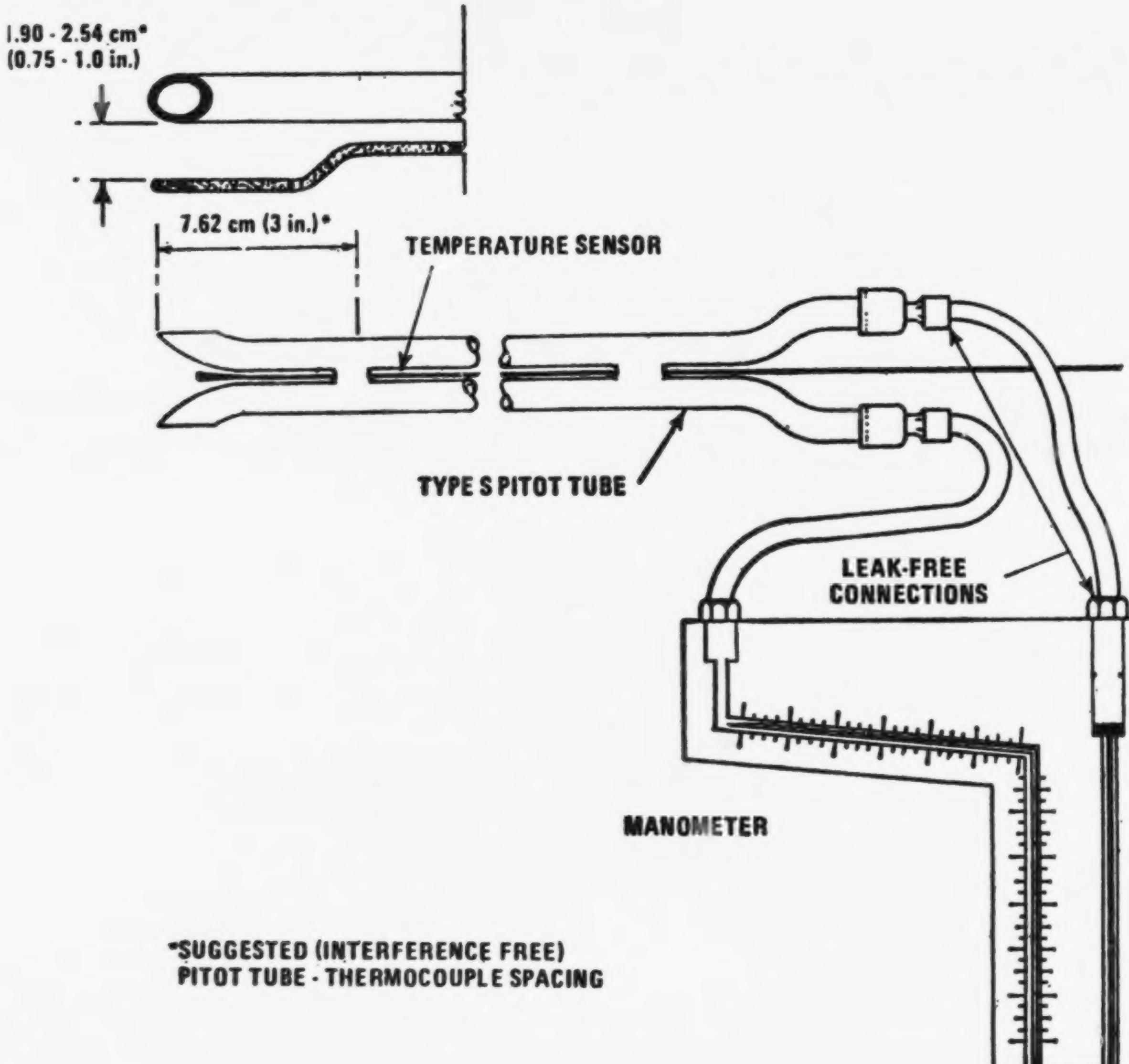

1.90 - 2.54 cm*
(0.75 - 1.0 in.)
7.62 cm (3 in.)*
TEMPERATURE SENSOR
TYPE S PITOT TUBE
LEAK-FREE
CONNECTIONS
MANOMETER
*SUGGESTED (INTERFERENCE FREE)
PITOT TUBE - THERMOCOUPLE SPACING

Figure 2-1. Type S pitot tube manometer assembly.

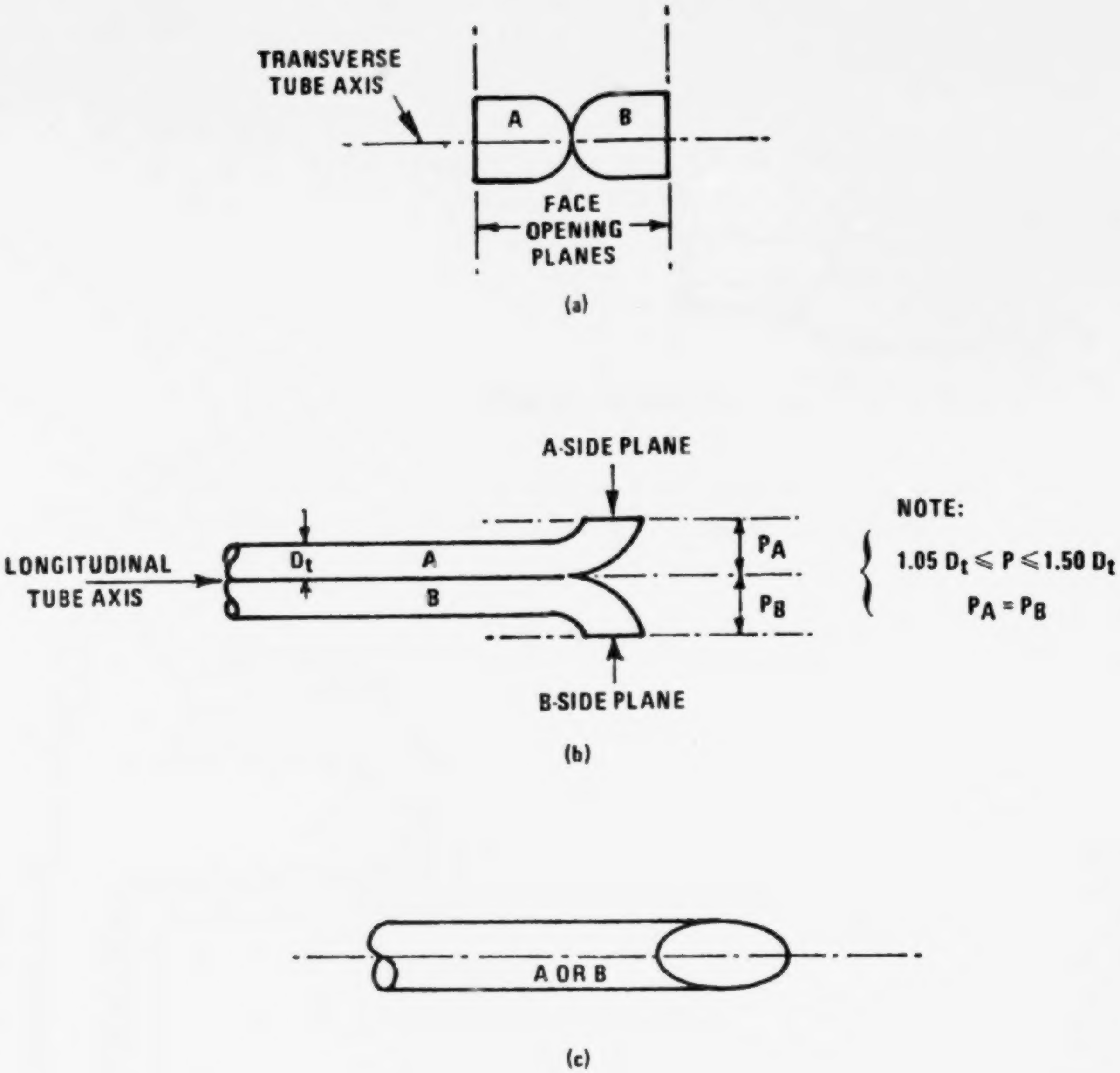

TRANSVERSE TUBE AXIS
A
B
FACE OPENING PLANES
(a)
A-SIDE PLANE
NOTE:
$1.05\,D_t \leq P \leq 1.50\,D_t$
$P_A = P_B$
LONGITUDINAL TUBE AXIS
$D_t$
A
B
$P_A$
$P_B$
B-SIDE PLANE
(b)
A OR B
(c)

Figure 2-2. Properly constructed Type S pitot tube, shown in: (a) end view; face opening planes perpendicular to transverse axis; (b) top view; face opening planes parallel to longitudinal axis; (c) side view; both legs of equal length and centerlines coincident, when viewed from both sides. Baseline coefficient values of 0.84 may be assigned to pitot tubes constructed this way.

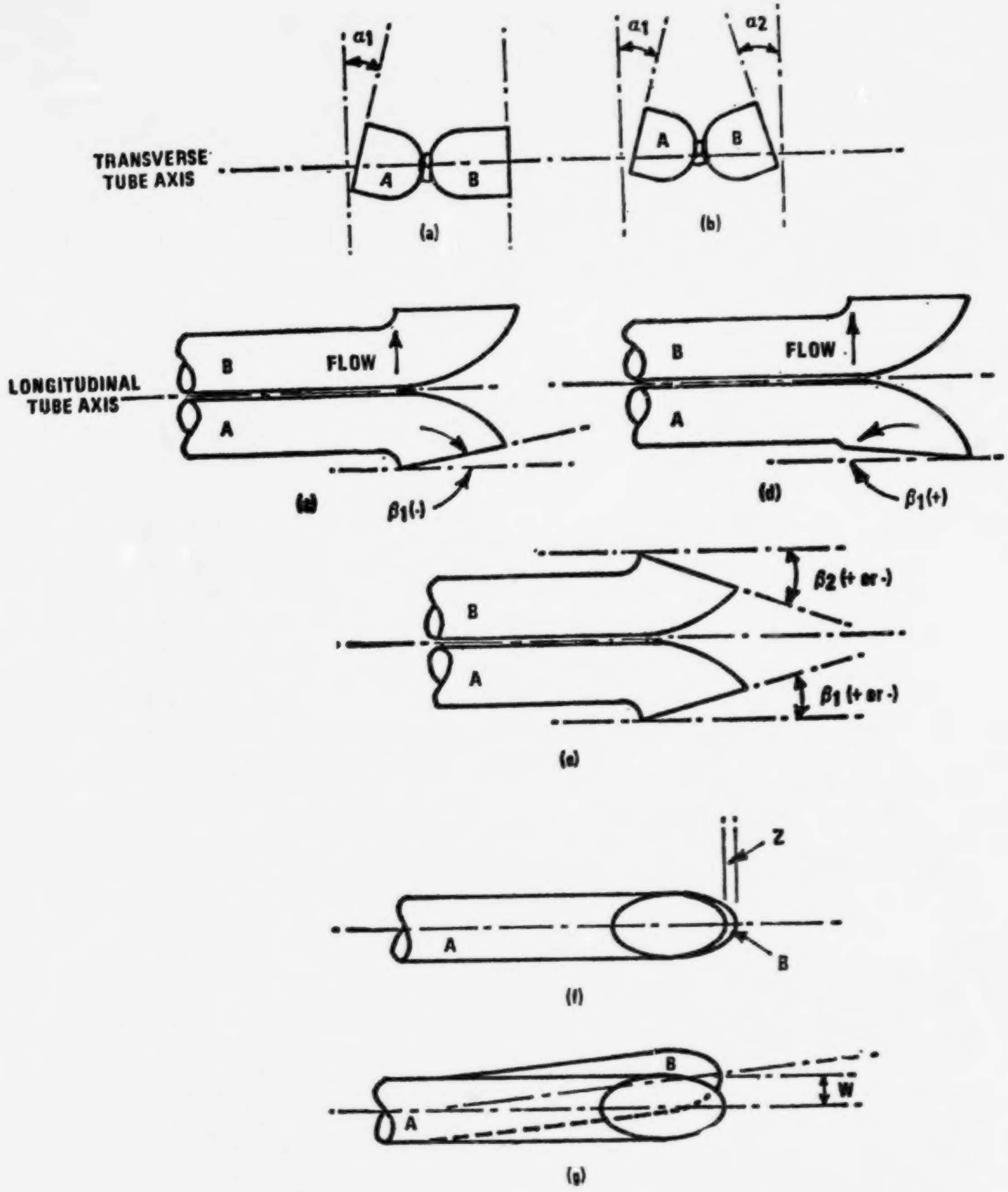

α1
α2
TRANSVERSE TUBE AXIS
A
B
(a)
(b)
LONGITUDINAL TUBE AXIS
FLOW
(c)
β1(-)
(d)
β1(+)
β2 (+ or -)
β1 (+ or -)
(e)
Z
(f)
W
(g)

**Figure 2-3.** Types of face-opening misalignment that can result from field use or improper construction of Type S pitot tubes. These will not affect the baseline value of $\overline{C}p(s)$ so long as $\alpha_1$ and $\alpha_2 < 10^{\circ}$, $\beta_1$ and $\beta_2 < 5^{\circ}$. $z < 0.32$ cm (1/8 in.) and $w < 0.08$ cm (1/32 in.) (citation 11 in Section 6).

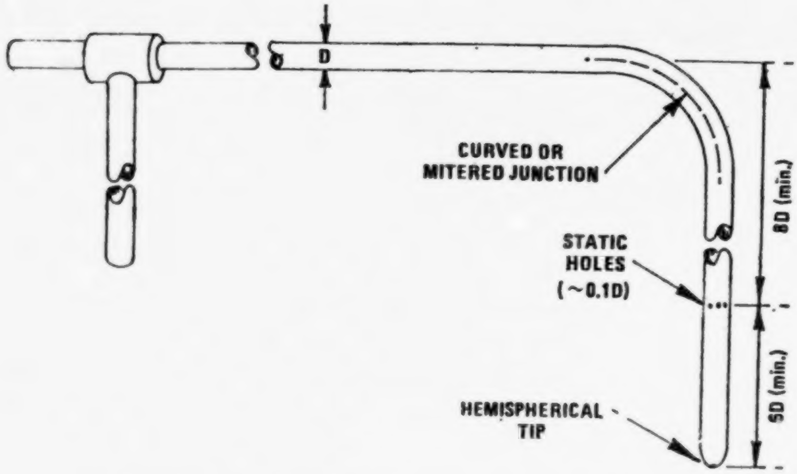

D
CURVED OR MITERED JUNCTION
8D (min.)
STATIC HOLES (~0.1D)
5D (min.)
HEMISPHERICAL TIP

Figure 2-4. Standard pitot tube design specifications.

Section 6) may be used. Pitot tubes designed according to these specifications will have baseline coefficients of about 0.99 ± 0.01.

2.7.1 Hemispherical (shown in Figure 2-4), ellipsoidal, or conical tip.

2.7.2 A minimum of six diameters straight run (based upon $D$, the external diameter of the tube) between the tip and the static pressure holes.

2.7.3 A minimum of eight diameters straight run between the static pressure holes and the centerline of the external tube, following the 90 degree bend.

2.7.4 Static pressure holes of equal size (approximately 0.1 $D$), equally spaced in a piezometer ring configuration.

2.7.5 Ninety degree bend, with curved or mitered junction.

2.8 Differential Pressure Gauge for Type S Pitot Tube Calibration. An inclined manometer or equivalent is used. If the single-velocity calibration technique is employed (see Section 4.1.2.3), the calibration differential pressure gauge shall be readable to the nearest 0.13 mm $H_2O$ (0.005 in. $H_2O$). For multivelocity calibrations, the gauge shall be readable to the nearest 0.13 mm $H_2O$ (0.005 in $H_2O$) for $\Delta p$ values between 1.3 and 25 mm $H_2O$ (0.05 and 1.0 in. $H_2O$), and to the nearest 1.3 mm $H_2O$ (0.05 in. $H_2O$) for $\Delta p$ values above 25 mm $H_2O$ (1.0 in. $H_2O$). A special, more sensitive gauge will be required to read $\Delta p$ values below 1.3 mm $H_2O$ [0.05 in. $H_2O$] (see Citation 18 in Section 6).

### 3. *Procedure*

3.1 Set up the apparatus as shown in Figure 2-1. Capillary tubing or surge tanks installed between the manometer and pitot tube may be used to dampen $\Delta p$ fluctuations. It is recommended, but not required, that a pretest leak-check be conducted, as follows: (1) blow through the pitot impact opening until at least 7.6 cm (3 in.) $H_2O$ velocity pressure registers on the manometer; then, close off the impact opening. The pressure shall remain stable for at least 15 seconds; (2) do the same for the static pressure side, except using suction to obtain the minimum of 7.6 cm (3 in.) $H_2O$. Other leak-check procedures, subject to the approval of the Administrator, may be used.

3.2 Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse. Record all necessary data as shown in the example data sheet (Figure 2-5).

3.3 Measure the velocity head and temperature at the traverse points specified by Method 1. Ensure that the proper differential pressure gauge is being used for the range of $\Delta p$ values encountered (see Section 2.2). If it is necessary to change to a more sensitive gauge, do so, and remeasure the $\Delta p$ and temperature readings at each traverse point. Conduct a post-test leak-check (mandatory), as described in Section 3.1 above, to validate the traverse run.

3.4 Measure the static pressure in the stack. One reading is usually adequate.

3.5 Determine the atmospheric pressure.

3.6 Determine the stack gas dry molecular weight. For combustion processes or processes that emit essentially $CO_2$, $O_2$, CO, and $N_2$, use Method 3. For processes emitting essentially air, an analysis need not be conducted; use a dry molecular weight of 29.0. For other processes, other methods, subject to the approval of the Administrator, must be used.

3.7 Obtain the moisture content from Reference Method 4 (or equivalent) or from Method 5.

3.8 Determine the cross-sectional area of the stack or duct at the sampling location. Whenever possible, physically measure the stack dimensions rather than using blueprints.

### 4 *Calibration*

4.1 Type S Pitot Tube. Before its initial use, carefully examine the Type S pitot tube in top, side, and end views to verify that the face openings of the tube are aligned within the specifications illustrated in Figure 2-2 or 2-3. The pitot tube shall not be used if it fails to meet these alignment specifications.

After verifying the face opening alignment, measure and record the following dimensions of the pitot tube: (a) the external tubing diameter (dimension $D_t$, Figure 2-2b); and (b) the base-to-opening plane distances (dimensions $P_A$ and $P_B$, Figure 2-2b). If $D_t$ is between 0.48 and 0.95 cm (3/16 and 3/8 in.) and if $P_A$ and $P_B$ are equal and between 1.05 and 1.50 $D_t$, there are two possible options: (1) the pitot tube may be calibrated according to the procedure outlined in Sections 4.1.2 through 4.1.5 below, or (2) a baseline (isolated tube) coefficient value of 0.84 may be assigned to the pitot tube. Note, however, that if the pitot tube is part of an assembly, calibration may still be required, despite knowledge of the baseline coefficient value (see Section 4.1.1).

If $D_t$, $P_A$, and $P_B$ are outside the specified limits, the pitot tube must be calibrated as outlined in 4.1.2 through 4.1.5 below.

4.1.1 Type S Pitot Tube Assemblies. During sample and velocity traverses, the isolated Type S pitot tube is not always used; in many instances, the pitot tube is used in combination with other source-sampling components (thermocouple, sampling probe, nozzle) as part of an "assembly." The presence of other sampling components can sometimes affect the baseline value of the Type S pitot tube coefficient (Citation 9 in Section 6); therefore an assigned (or otherwise known) baseline coefficient value may or may not be valid for a given assembly. The baseline and assembly coefficient values will be identical only when the relative placement of the components in the assembly is such that aerodynamic interference effects are eliminated. Figures 2-6 through 2-8 illustrate interference-free component arrangements for Type S pitot tubes having external tubing diameters between 0.48 and 0.95 cm (3/16 and 3/8 in.). Type S pitot tube assemblies that fail to meet any or all of the specifications of Figures 2-6 through 2-8 shall be calibrated according to the procedure outlined in Sections 4.1.2 through 4.1.5 below, and prior to calibration, the values of the intercomponent spacings (pitot-nozzle, pitot-thermocouple, pitot-probe sheath) shall be measured and recorded.

NOTE.—Do not use any Type S pitot tube assembly which is constructed such that the impact pressure opening plane of the pitot tube is below the entry plane of the nozzle (see Figure 2-6b).

4.1.2 Calibration Setup. If the Type S pitot tube is to be calibrated, one leg of the tube shall be permanently marked A, and the other, B. Calibration shall be done in a flow system having the following essential design features:

4.1.2.1 The flowing gas stream must be confined to a duct of definite cross-sectional area, either circular or rectangular. For circular cross-sections, the minimum duct diameter shall be 30.5 cm (12 in.); for rectangular cross-sections, the width (shorter side) shall be at least 25.4 cm (10 in.).

4.1.2.2 The cross-sectional area of the calibration duct must be constant over a distance of 10 or more duct diameters. For a rectangular cross-section, use an equivalent diameter, calculated from the following equation, to determine the number of duct diameters:

$$D_e = \frac{2\,LW}{(L+W)}$$

Equation 2-1

where:

$D_e$ = Equivalent diameter

$L$ = Length

$W$ = Width

To ensure the presence of stable, fully developed flow patterns at the calibration site, or "test section," the site must be located at least eight diameters downstream and two diameters upstream from the nearest disturbances.

NOTE.—The eight- and two-diameter criteria are not absolute; other test section locations may be used (subject to approval of the Administrator), provided that the flow at the test site is stable and demonstrably parallel to the duct axis.

4.1.2.3 The flow system shall have the capacity to generate a test-section velocity around 915 m/min (3,000 ft/min). This velocity must be constant with time to guarantee steady flow during calibration. Note that Type S pitot tube coefficients obtained by single-velocity calibration at 915 m/min (3,000 ft/min) will generally be valid to within ±3 percent for the measurement of velocities above 305 m/min (1,000 ft/min) and to within ±5 to 6 percent for the measurement of velocities between 180 and 305 m/min (600 and 1,000 ft/min). If a more precise correlation between $C_p$ and velocity is desired, the flow system shall have the capacity to generate at least four distinct, time-invariant test-section velocities covering the velocity range from 180 to 1,525 m/min (600 to 5,000 ft/min), and calibration data shall be taken at regular velocity intervals over this range (see Citations 9 and 14 in Section 6 for details).

4.1.2.4 Two entry ports, one each for the standard and Type S pitot tubes, shall be cut in the test section; the standard pitot entry port shall be located slightly downstream of the Type S port, so that the standard and Type S impact openings will lie in the same cross-sectional plane during calibration. To facilitate alignment of the pitot tubes during calibration, it is advisable that the test section be constructed of plexiglas or some other transparent material.

4.1.3 Calibration Procedure. Note that this procedure is a general one and must not be used without first referring to the special considerations presented in Section 4.1.5. Note also that this procedure applies only to single-velocity calibration. To obtain calibration data for the A and B sides of the Type S pitot tube, proceed as follows:

4.1.3.1 Make sure that the manometer is properly filled and that the oil is free from contamination and is of the proper density. Inspect and leak-check all pitot lines; repair or replace if necessary.

4.1.3.2 Level and zero the manometer. Turn on the fan and allow the flow to stabilize. Seal the Type S entry port.

4.1.3.3 Ensure that the manometer is level and zeroed. Position the standard pitot tube at the calibration point (determined as outlined in Section 4.1.5.1), and align the tube so that its tip is pointed directly into the flow. Particular care should be taken in aligning the tube to avoid yaw and pitch angles. Make sure that the entry port surrounding the tube is properly sealed.

4.1.3.4 Read $\Delta p_{std}$ and record its value in a data table similar to the one shown in Figure 2-9. Remove the standard pitot tube from the duct and disconnect it from the manometer. Seal the standard entry port.

4.1.3.5 Connect the Type S pitot tube to the manometer. Open the Type S entry port. Check the manometer level and zero. Insert and align the Type S pitot tube so that its A side impact opening is at the same point as was the standard pitot tube and is pointed directly into the flow. Make sure that the entry port surrounding the tube is properly sealed.

**PLANT** ____________________

**DATE** ____________ **RUN NO.** ____________

**STACK DIAMETER OR DIMENSIONS, m(in.)** ____________

**BAROMETRIC PRESSURE, mm Hg (in. Hg)** ____________

**CROSS SECTIONAL AREA, $m^2(ft^2)$** ____________

**OPERATORS** ____________

**PITOT TUBE I.D. NO.** ____________

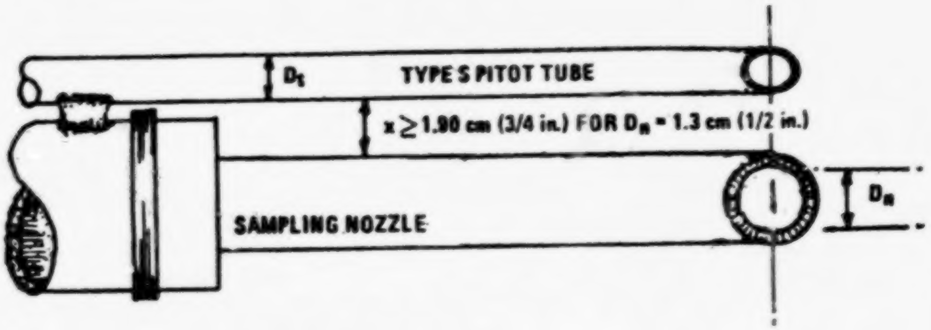
**AVG. COEFFICIENT, $C_p$ =** ____________

**LAST DATE CALIBRATED** ____________

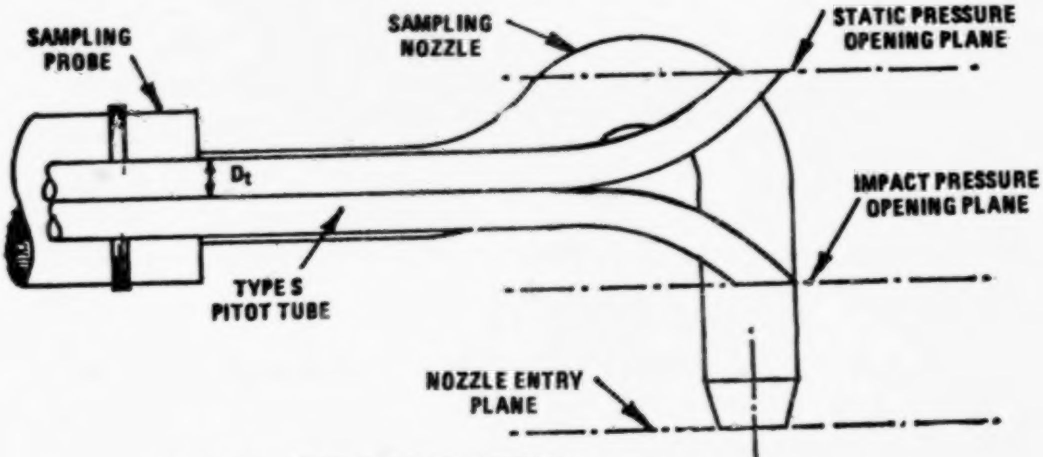
**SCHEMATIC OF STACK CROSS SECTION**

| Traverse Pt. No. | Vel. Hd., $\Delta p$ mm (in.) $H_2O$ | Stack Temperature | | $P_g$ mm Hg (in.Hg) | $\sqrt{\Delta p}$ |
|---|---|---|---|---|---|
| | | $t_s$, °C (°F) | $T_s$, °K (°R) | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | Average | | | |

Figure 2-5. Velocity traverse data.


D_t
TYPE S PITOT TUBE
x ≥ 1.90 cm (3/4 in.) FOR D_n = 1.3 cm (1/2 in.)
SAMPLING NOZZLE
D_n

A. BOTTOM VIEW; SHOWING MINIMUM PITOT-NOZZLE SEPARATION.


SAMPLING PROBE
SAMPLING NOZZLE
STATIC PRESSURE OPENING PLANE
D_t
IMPACT PRESSURE OPENING PLANE
TYPE S PITOT TUBE
NOZZLE ENTRY PLANE

B. SIDE VIEW; TO PREVENT PITOT TUBE FROM INTERFERING WITH GAS FLOW STREAMLINES APPROACHING THE NOZZLE, THE IMPACT PRESSURE OPENING PLANE OF THE PITOT TUBE SHALL BE EVEN WITH OR ABOVE THE NOZZLE ENTRY PLANE.

Figure 2-6. Proper pitot tube - sampling nozzle configuration to prevent aerodynamic interference; buttonhook - type nozzle; centers of nozzle and pitot opening aligned; $D_t$ between 0.48 and 0.95 cm (3/16 and 3/8 in.).

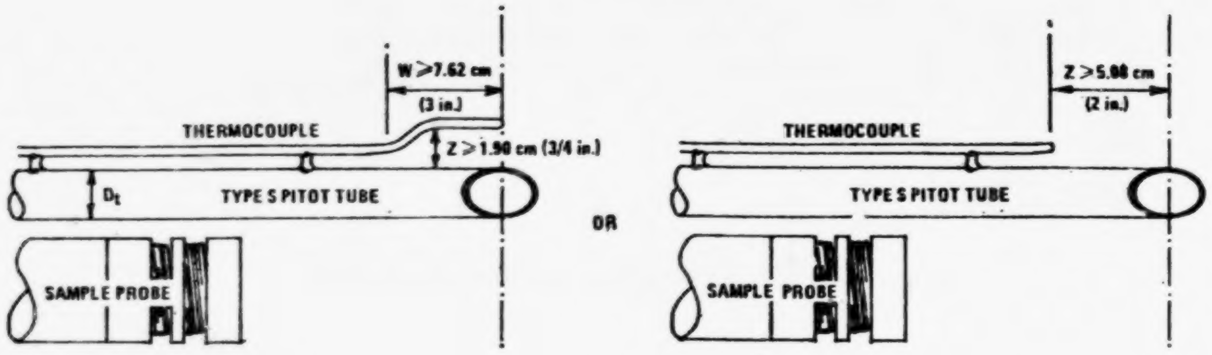

W ≥7.62 cm
(3 in.)
THERMOCOUPLE
Z ≥ 1.90 cm (3/4 in.)
$D_t$
TYPE S PITOT TUBE
SAMPLE PROBE
OR
Z ≥ 5.08 cm
(2 in.)
THERMOCOUPLE
TYPE S PITOT TUBE
SAMPLE PROBE

Figure 2-7. Proper thermocouple placement to prevent interference; $D_t$ between 0.48 and 0.95 cm (3/16 and 3/8 in.).

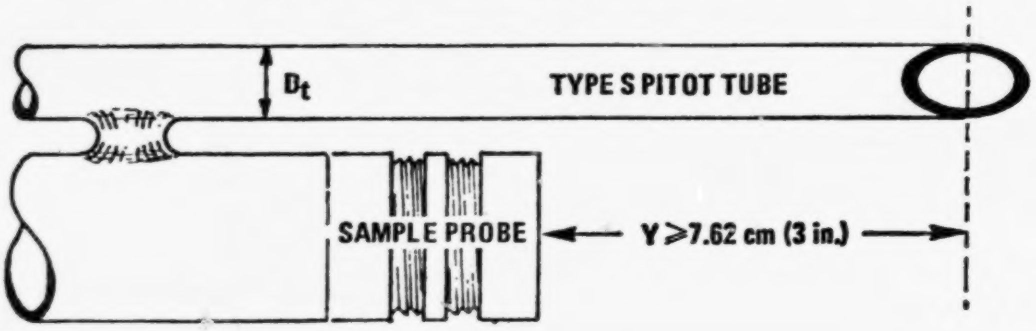

$D_t$
TYPE S PITOT TUBE
SAMPLE PROBE
Y ≥7.62 cm (3 in.)

Figure 2-8. Minimum pitot-sample probe separation needed to prevent interference; $D_t$ between 0.48 and 0.95 cm (3/16 and 3/8 in.).

**PITOT TUBE IDENTIFICATION NUMBER:** ____________________ **DATE:** ______________

**CALIBRATED BY:** ________________________________________________

| | "A" SIDE CALIBRATION | | | |
|---|---|---|---|---|
| RUN NO. | $\Delta p_{std}$ cm $H_2O$ (in. $H_2O$) | $\Delta p_{(s)}$ cm $H_2O$ (in. $H_2O$) | $C_{p(s)}$ | DEVIATION $C_{p(s)} - \bar{C}_p(A)$ |
| 1 | | | | |
| 2 | | | | |
| 3 | | | | |
| | | $\bar{C}_p$ (SIDE A) | | |

| | "B" SIDE CALIBRATION | | | |
|---|---|---|---|---|
| RUN NO. | $\Delta p_{std}$ cm $H_2O$ (in. $H_2O$) | $\Delta p_{(s)}$ cm $H_2O$ (in. $H_2O$) | $C_{p(s)}$ | DEVIATION $C_{p(s)} - \bar{C}_p(B)$ |
| 1 | | | | |
| 2 | | | | |
| 3 | | | | |
| | | $\bar{C}_p$ (SIDE B) | | |

$$\text{AVERAGE DEVIATION} = \sigma\,(\text{A OR B}) = \frac{\sum_{1}^{3} \left| C_p(s) - \bar{C}_p(\text{A OR B}) \right|}{3} \longleftarrow \text{MUST BE} \leq 0.01$$

$$\left| \bar{C}_p\,(\text{SIDE A}) - \bar{C}_p\,(\text{SIDE B}) \right| \longleftarrow \text{MUST BE} < 0.01$$

Figure 2-9. Pitot tube calibration data.

4.1.3.6 Read $\Delta p_s$ and enter its value in the data table. Remove the Type S pitot tube from the duct and disconnect it from the manometer.

4.1.3.7 Repeat steps 4.1.3.3 through 4.1.3.6 above until three pairs of $\Delta p$ readings have been obtained.

4.1.3.8 Repeat steps 4.1.3.3 through 4.1.3.7 above for the B side of the Type S pitot tube.

4.1.3.9 Perform calculations, as described in Section 4.1.4 below.

4.1.4 Calculations.

4.1.4.1 For each of the six pairs of $\Delta p$ readings (i.e., three from side A and three from side B) obtained in Section 4.1.3 above, calculate the value of the Type S pitot tube coefficient as follows:

$$C_{p(s)} = C_{p(std)} \sqrt{\frac{\Delta p_{std}}{\Delta p_s}}$$

Equation 2-2

**where:**

$C_{p(s)}$ = **Type S pitot tube coefficient**

$C_{p(std)}$ = **Standard pitot tube coefficient; use 0.99 if the coefficient is unknown and the tube is designed according to the criteria of Sections 2.7.1 to 2.7.5 of this method.**

$\Delta p_{std}$ = **Velocity head measured by the standard pitot tube, cm $H_2O$ (in. $H_2O$)**

$\Delta p_s$ = **Velocity head measured by the Type S pitot tube, cm $H_2O$ (in. $H_2O$)**

**4.1.4.2 Calculate $\overline{C}_p$ (side A), the mean A-side coefficient, and $\overline{C}_p$ (side B), the mean B-side coefficient; calculate the difference between these two average values.**

4.1.4.3 Calculate the deviation of each of the three A-side values of $C_{p(s)}$ from $\overline{C}_p$ (side A), and the deviation of each B-side value of $C_{p(s)}$ from $\overline{C}_p$ (side B). Use the following equation:

$$\text{Deviation} = C_{p(s)} - \overline{C}_p(\text{A or B})$$

Equation 2-3

4.1.4.4 Calculate $\sigma$, the average deviation from the mean, for both the A and B sides of the pitot tube. Use the following equation:

$$\sigma\,(\text{side A or B}) = \frac{\sum_{1}^{3} |C_{p(s)} - \overline{C}_p(A \text{ or } B)|}{3}$$

Equation 2-4

4.1.4.5 Use the Type S pitot tube only if the values of $\sigma$ (side A) and $\sigma$ (side B) are less than or equal to 0.01 and if the absolute value of the difference between $\overline{C}_p$ (A) and $\overline{C}_p$ (B) is 0.01 or less.

4.1.5 Special considerations.

4.1.5.1 Selection of calibration point.

4.1.5.1.1 When an isolated Type S pitot tube is calibrated, select a calibration point at or near the center of the duct, and follow the procedures outlined in Sections 4.1.3 and 4.1.4 above. The Type S pitot coefficients so obtained, i.e., $\overline{C}_p$ (side A) and $\overline{C}_p$ (side B), will be valid, so long as either: (1) the isolated pitot tube is used; or (2) the pitot tube is used with other components (nozzle, thermocouple, sample probe) in an arrangement that is free from aerodynamic interference effects (see Figures 2-6 through 2-8).

4.1.5.1.2 For Type S pitot tube-thermocouple combinations (without sample probe), select a calibration point at or near the center of the duct, and follow the procedures outlined in Sections 4.1.3 and 4.1.4 above. The coefficients so obtained will be valid so long as the pitot tube-thermocouple combination is used by itself or with other components in an interference-free arrangement (Figures 2-6 and 2-8).

4.1.5.1.3 For assemblies with sample probes, the calibration point should be located at or near the center of the duct; however, insertion of a probe sheath into a small duct may cause significant cross-sectional area blockage and yield incorrect coefficient values (Citation 9 in Section 6). Therefore, to minimize the blockage effect, the calibration point may be a few inches off-center if necessary. The actual blockage effect will be negligible when the theoretical blockage, as determined by a projected-area model of the probe sheath, is 2 percent or less of the duct cross-sectional area for assemblies without external sheaths (Figure 2-10a), and 3 percent or less for assemblies with external sheaths (Figure 2-10b).

4.1.5.2 For those probe assemblies in which pitot tube-nozzle interference is a factor (i.e., those in which the pitot-nozzel separation distance fails to meet the specification illustrated in Figure 2-6a), the value of $C_{p(s)}$ depends upon the amount of free-space between the tube and nozzle, and therefore is a function of nozzle size. In these instances, separate calibrations shall be performed with each of the commonly used nozzle sizes in place. Note that the single-velocity calibration technique is acceptable for this purpose, even though the larger nozzle sizes (>0.635 cm or ¼ in.) are not ordinarily used for isokinetic sampling at velocities around 915 m/min (3,000 ft/min), which is the calibration velocity; note also that it is not necessary to draw an isokinetic sample during calibration (see Citation 19 in Section 6).

4.1.5.3 For a probe assembly constructed such that its pitot tube is always used in the same orientation, only one side of the pitot tube need be calibrated (the side which will face the flow). The pitot tube must still meet the alignment specifications of Figure 2-2 or 2-3, however, and must have an average deviation ($\sigma$) value of 0.01 or less (see Section 4.1.4.4).

4.1.6 Field Use and Recalibration.

4.1.6.1 Field Use.

4.1.6.1.1 When a Type S pitot tube (isolated tube or assembly) is used in the field, the appropriate coefficient value (whether assigned or obtained by calibration) shall be used to perform velocity calculations. For calibrated Type S pitot tubes, the A side coefficient shall be used when the A side of the tube faces the flow, and the B side coefficient shall be used when the B side faces the flow; alternatively, the arithmetic average of the A and B side coefficient values may be used, irrespective of which side faces the flow.

4.1.6.1.2 When a probe assembly is used to sample a small duct (12 to 36 in. in diameter), the probe sheath sometimes blocks a significant part of the duct cross-section, causing a reduction in the effective value of $\overline{C}_{p(s)}$. Consult Citation 9 in Section 6 for details. Conventional pitot-sampling probe assemblies are not recommended for use in ducts having inside diameters smaller than 12 inches (Citation 16 in Section 6).

4.1.6.2 Recalibration.

4.1.6.2.1 Isolated Pitot Tubes. After each field use, the pitot tube shall be carefully reexamined in top, side, and end views. If the pitot face openings are still aligned within the specifications illustrated in Figure 2-2 or 2-3, it can be assumed that the baseline coefficient of the pitot tube has not changed. If, however, the tube has been damaged to the extent that it no longer meets the specifications of Figure 2-2 or 2-3, the damage shall either be repaired to restore proper alignment of the face openings or the tube shall be discarded.

4.1.6.2.2 Pitot Tube Assemblies. After each field use, check the face opening alignment of the pitot tube, as in Section 4.1.6.2.1; also, remeasure the intercomponent spacings of the assembly. If the intercomponent spacings have not changed and the face opening alignment is acceptable, it can be assumed that the coefficient of the assembly has not changed. If the face opening alignment is no longer within the specifications of Figures 2-2 or 2-3, either repair the damage or replace the pitot tube (calibrating the new assembly, if necessary). If the intercomponent spacings have changed, restore the original spacings or recalibrate the assembly.

4.2 Standard pitot tube (if applicable). If a standard pitot tube is used for the velocity traverse, the tube shall be constructed according to the criteria of Section 2.7 and shall be assigned a baseline coefficient value of 0.99. If the standard pitot tube is used as part of an assembly, the tube shall be in an interference-free arrangement (subject to the approval of the Administrator).

4.3 Temperature Gauges. After each field use, calibrate dial thermometers, liquid-filled bulb thermometers, thermocouple-potentiometer systems, and other gauges at a temperature within 10 percent of the average absolute stack temperature. For temperatures up to 405° C (761° F), use an ASTM mercury-in-glass reference thermometer, or equivalent, as a reference; alternatively, either a reference thermocouple and potentiometer (calibrated by NBS) or thermometric fixed points, e.g., ice bath and boiling water (corrected for barometric pressure) may be used. For temperatures above 405° C (761° F), use an NBS-calibrated reference thermocouple-potentiometer system or an alternate reference, subject to the approval of the Administrator.

If, during calibration, the absolute temperatures measured with the gauge being calibrated and the reference gauge agree within 1.5 percent, the temperature data taken in the field shall be considered valid. Otherwise, the pollutant emission test shall either be considered invalid or adjustments (if appropriate) of the test results shall be made, subject to the approval of the Administrator.

4.4 Barometer. Calibrate the barometer used against a mercury barometer.

## 5. *Calculations*

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

5.1 Nomenclature.

$A$ = Cross-sectional area of stack, $m^2$ ($ft^2$).

$B_{ws}$ = Water vapor in the gas stream (from Method 5 or Reference Method 4), proportion by volume.

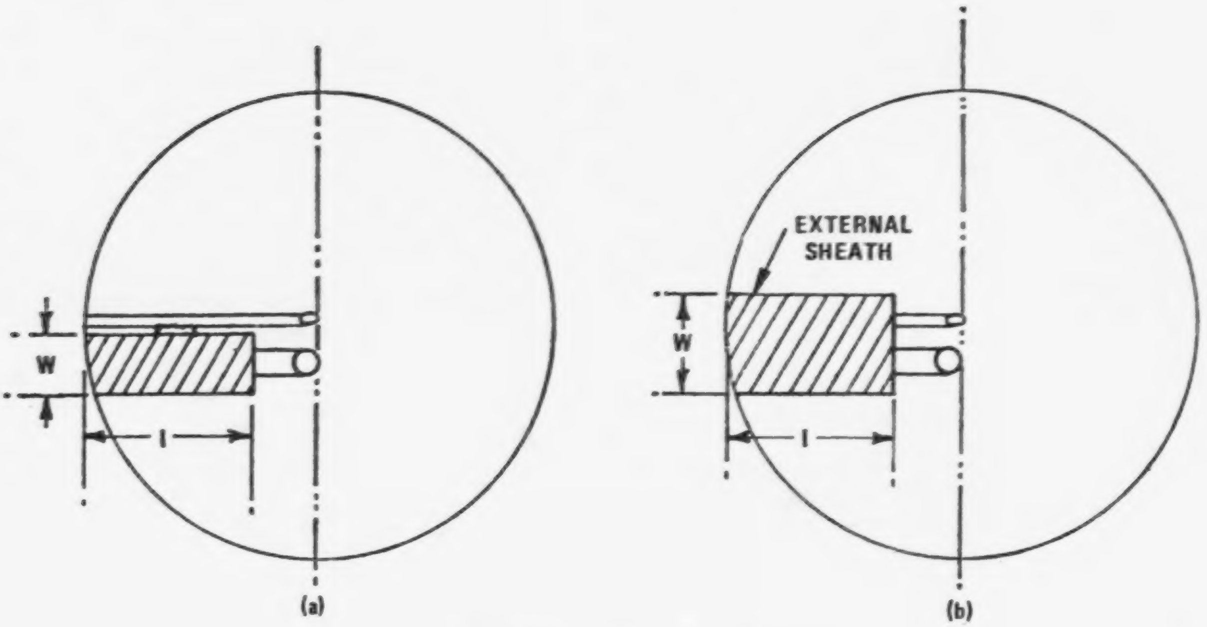
$C_p$ = Pitot tube coefficient, dimensionless.

$K_p$ = Pitot tube constant,

$$34.97 \frac{\text{m}}{\text{sec}} \left[\frac{(g/g\text{-mole})(\text{mm Hg})}{(^\circ\text{K})(\text{mm } H_2O)}\right]^{1/2}$$

for the metric system and

$$85.49 \frac{\text{ft}}{\text{sec}} \left[\frac{(\text{lb/lb-mole})(\text{in. Hg})}{(^\circ\text{R})(\text{in. } H_2O)}\right]^{1/2}$$


W
l
(a)
EXTERNAL SHEATH
W
l
(b)

$$\text{ESTIMATED SHEATH BLOCKAGE (\%)} = \left[\frac{l \times W}{\text{DUCT AREA}}\right] \times 100$$

Figure 2-10. Projected-area models for typical pitot tube assemblies.

for the English system.

$M_d$=Molecular weight of stack gas, dry basis (see Section 3.6) g/g-mole (lb/lb-mole).

$M_s$=Molecular weight of stack gas, wet basis, g/g-mole (lb/lb-mole).

$$=M_d\,(1-B_{ws})+18.0\,B_{ws} \qquad \text{Equation 2-5}$$

$P_{bar}$=Barometric pressure at measurement site, mm Hg (in. Hg).

$P_g$=Stack static pressure, mm Hg (in. Hg).

$P_s$=Absolute stack gas pressure, mm Hg (in. Hg).

$$=P_{bar}+P_g \qquad \text{Equation 2-6}$$

$P_{std}$=Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

$Q_{sd}$=Dry volumetric stack gas flow rate corrected to standard conditions, dscm/hr (dscf/hr).

$t_s$=Stack temperature, °C (°F).

$T_s$=Absolute stack temperature, °K (°R).

$$=273+t_s \text{ for metric} \qquad \text{Equation 2-7}$$

$$=460+t_s \text{ for English} \qquad \text{Equation 2-8}$$

$T_{std}$=Standard absolute temperature, 293°K (528° R)

$v_s$=Average stack gas velocity, m/sec (ft/sec).

$\Delta p$=Velocity head of stack gas, mm $H_2O$ (in. $H_2O$).

3,600=Conversion factor, sec/hr.

18.0=Molecular weight of water, g/g-mole (lb-lb-mole).

5.2 Average stack gas velocity.

$$v_s=K_pC_p(\sqrt{\Delta p})_{avg}\sqrt{\frac{T_{s(avg)}}{P_sM_s}}$$

Equation 2-9

5.3 Average stack gas dry volumetric flow rate.

$$Q_{sd}=3{,}600(1-B_{ws})v_sA\left(\frac{T_{std}}{T_{s(avg)}}\right)\left(\frac{P_s}{P_{std}}\right)$$

Equation 2-10

6. ***Bibliography***

1. Mark, L. S. Mechanical Engineers' Handbook. New York, McGraw-Hill Book Co., Inc. 1951.

2. Perry, J. H. Chemical Engineers' Handbook. New York. McGraw-Hill Book Co., Inc. 1960.

3. Shigehara, R. T., W. F. Todd, and W. S. Smith. Significance of Errors in Stack Sampling Measurements. U.S. Environmental Protection Agency, Research Triangle Park, N.C. (Presented at the Annual Meeting of the Air Pollution Control Association, St. Louis, Mo., June 14-19, 1970.)

4. Standard Method for Sampling Stacks for Particulate Matter. In: 1971 Book of ASTM Standards, Part 23. Philadelphia, Pa. 1971. ASTM Designation D-2928-71.

5. Vennard, J. K. Elementary Fluid Mechanics. New York. John Wiley and Sons, Inc. 1947.

6. Fluid Meters—Their Theory and Application. American Society of Mechanical Engineers, New York, N.Y. 1971.

7. ASHRAE Handbook of Fundamentals. 1972. p. 208.

8. Annual Book of ASTM Standards, Part 26. 1974. p. 648.

9. Vollaro, R. F. Guidelines for Type S Pitot Tube Calibration. U.S. Environmental Protection Agency, Research Triangle Park, N.C. (Presented at 1st Annual Meeting, Source Evaluation Society, Dayton, Ohio, September 18, 1975.)

10. Vollaro, R. F. A Type S Pitot Tube Calibration Study. U.S. Environmental Protection Agency, Emission Measurement Branch, Research Triangle Park, N.C. July 1974.

11. Vollaro, R. F. The Effects of Impact Opening Misalignment on the Value of the Type S Pitot Tube Coefficient. U.S. Environmental Protection Agency, Emission Measurement Branch, Research Triangle Park, N.C. October 1976.

12. Vollaro, R. F. Establishment of a Baseline Coefficient Value for Properly Constructed Type S Pitot Tubes. U.S. Environmental Protection Agency, Emission Measurement Branch, Research Triangle Park, N.C. November 1976.

13. Vollaro, R. F. An Evaluation of Single-Velocity Calibration Techniques as a Means of Determining Type S Pitot Tube Coefficients. U.S. Environmental Protection Agency, Emission Measurement Branch, Research Triangle Park, N.C. August 1975.

14. Vollaro, R. F. The Use of Type S Pitot Tubes for the Measurement of Low Velocities. U.S. Environmental Protection Agency, Emission Measurement Branch, Research Triangle Park, N.C. November 1976.

15. Smith, Marvin L. Velocity Calibration of EPA Type Source Sampling Probe. United Technologies Corporation, Pratt and Whitney Aircraft Division, East Hartford, Conn. 1975.

16. Vollaro, R. F. Recommended Procedure for Sample Traverses in Ducts Smaller than 12 Inches in Diameter. U.S. Environmental Protection Agency, Emission Measurement Branch, Research Triangle Park, N.C. November 1976.

17. Ower, E. and R. C. Pankhurst. The Measurement of Air Flow, 4th Ed., London, Pergamon Press. 1966.

18. Vollaro, R. F. A survey of Commercially Available Instrumentation for the Measurement of Low-Range Gas Velocities. U.S. Environmental Protection Agency, Emission Measurement Branch, Research Triangle Park, N.C. November 1976. (Unpublished Paper)

19. Gnyp, A. W., C. C. St. Pierre, D. S. Smith, D. Mozzon, and J. Steiner. An Experimental Investigation of the Effect of Pitot Tube-Sampling Probe Configurations on the Magnitude of the S Type Pitot Tube Coefficient for Commercially Available Source Sampling Probes. Prepared by the University of Windsor for the Ministry of the Environment, Toronto, Canada. February 1975.

# METHOD 3

## GAS ANALYSIS FOR CARBON DIOXIDE, EXCESS AIR, AND DRY MOLECULAR WEIGHT

### 1. *Principle and Applicability*

1.1 **Principle.** A gas sample is extracted from a stack, by one of the following methods: (1) single-point, grab sampling; (2) single-point, integrated sampling; or (3) multi-point, integrated sampling. The gas sample is analyzed for percent carbon dioxide ($CO_2$), percent oxygen ($O_2$), and, if necessary, percent carbon monoxide (CO). If a dry molecular weight determination is to be made, either an Orsat or a Fyrite [1] analyzer may be used for the analysis; for excess air or emission rate correction factor determination, an Orsat analyzer must be used.

1.2 Applicability. This method is applicable for determining $CO_2$ and $O_2$ concentrations, excess air, and dry molecular weight of a sample from a gas stream of a fossil-fuel combustion process. The method may also be applicable to other processes where it has been determined that compounds other than $CO_2$, $O_2$, CO, and nitrogen ($N_2$) are not present in concentrations sufficient to affect the results.

Other methods, as well as modifications to the procedure described herein, are also applicable for some or all of the above determinations. Examples of specific methods and modifications include: (1) a multi-point sampling method using an Orsat analyzer to analyze individual grab samples obtained at each point; (2) a method using $CO_2$ or $O_2$ and stoichiometric calculations to determine dry molecular weight and excess air; (3) assigning a value of 30.0 for dry molecular weight, in lieu of actual measurements, for processes burning natural gas, coal, or oil. These methods and modifications may be used, but are subject to the approval of the Administrator.

### 2. *Apparatus*

As an alternative to the sampling apparatus and systems described herein, other sampling systems (e.g., liquid displacement) may be used provided such systems are capable of obtaining a representative sample and maintaining a constant sampling rate, and are otherwise capable of yielding acceptable results. Use of such systems is subject to the approval of the Administrator.

2.1 Grab Sampling (Figure 3-1).

2.1.1 Probe. The probe should be made of stainless steel or borosilicate glass tubing and should be equipped with an in-stack or out-stack filter to remove particulate matter (a plug of glass wool is satisfactory for this purpose). Any other material inert to $O_2$, $CO_2$, CO, and $N_2$ and resistant to temperature at sampling conditions may be used for the probe; examples of such material are aluminum, copper, quartz glass and Teflon.

2.1.2 Pump. A one-way [illegible] bulb, or equivalent, is used to transport the [illegible] sample to the analyzer.

2.2 Integrated Sampling (Figure 3-2).

2.2.1 Probe. A probe such as that described in Section 2.1.1 is suitable.

2.2.2 **Condenser.** An air-cooled or water-cooled condenser, or other condenser that will not remove $O_2$, $CO_2$, CO, and $N_2$, may be used to remove excess moisture which would interfere with the operation of the pump and flow meter.

2.2.3 **Valve.** A needle valve is used to adjust sample gas flow rate.

2.2.4 **Pump.** A leak-free, diaphragm-type pump, or equivalent, is used to transport sample gas to the flexible bag. Install a small surge tank between the pump and rate meter to eliminate the pulsation effect of the diaphragm pump on the rotameter.

2.2.5 **Rate Meter.** The rotameter, or equivalent rate meter, used should be capable of measuring flow rate to within ±2 percent of the selected flow rate. A flow rate range of 500 to 1000 $cm^3/min$ is suggested.

2.2.6 **Flexible Bag.** Any leak-free plastic (e.g., Tedlar, Mylar, Teflon) or plastic-coated aluminum (e.g., aluminized Mylar) bag, or equivalent, having a capacity consistent with the selected flow rate and time length of the test run, may be used. A capacity in the range of 55 to 90 liters is suggested.

To leak-check the bag, connect it to a water manometer and pressurize the bag to 5 to 10 cm $H_2O$ (2 to 4 in. $H_2O$). Allow to stand for 10 minutes. Any displacement in the water manometer indicates a leak. An alternative leak-check method is to pressurize the bag to 5 to 10 cm $H_2O$ (2 to 4 in. $H_2O$) and allow to stand overnight. A deflated bag indicates a leak.

2.2.7 Pressure Gauge. A water-filled U-tube manometer, or equivalent, of about 28 cm (12 in.) is used for the flexible bag leak-check.

2.2.8 Vacuum Gauge. A mercury manometer, or equivalent, of at least 760 mm Hg (30 in. Hg) is used for the sampling train leak-check.

2.3 **Analysis.** For Orsat and Fyrite analyzer maintenance and operation procedures, follow the instructions recommended by the manufacturer, unless otherwise specified herein.

2.3.1 Dry Molecular Weight Determination. An Orsat analyzer or Fyrite type combustion gas analyzer may be used.

2.3.2 Emission Rate Correction Factor or Excess Air Determination. An Orsat analyzer must be used. For low $CO_2$ (less than 4.0 percent) or high $O_2$ (greater than 15.0 percent) concentrations, the measuring burette of the Orsat must have at least 0.1 percent subdivisions.

### 3. *Dry Molecular Weight Determination*

Any of the three sampling and analytical procedures described below may be used for determining the dry molecular weight.

3.1 Single-Point, Grab Sampling and Analytical Procedure.

3.1.1 The sampling point in the duct shall either be at the centroid of the cross section or at a point no closer to the walls than 1.00 m (3.3 ft), unless otherwise specified by the Administrator.

3.1.2 Set up the equipment as shown in Figure 3-1, making sure all connections ahead of the analyzer are tight and leak-free. If an Orsat analyzer is used, it is recommended that the analyzer be leaked-checked by following the procedure in Section 5; however, the leak-check is optional.

3.1.3 Place the probe in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Draw a sample into the analyzer and immediately analyze it for percent $CO_2$ and percent $O_2$. Determine the percentage of the gas that is $N_2$ and CO by subtracting the sum of the percent $CO_2$ and percent $O_2$ from 100 percent. Calculate the dry molecular weight as indicated in Section 6.3.

3.1.4 Repeat the sampling, analysis, and calculation procedures, until the dry molecular weights of any three grab samples differ from their mean by no more than 0.3 g/g-mole (0.3 lb/lb-mole). Average these three molecular weights, and report the results to the nearest 0.1 g/g-mole (lb/lb-mole).

3.2 Single-Point, Integrated Sampling and Analytical Procedure.

3.2.1 The sampling point in the duct shall be located as specified in Section 3.1.1.

3.2.2 Leak-check (optional) the flexible bag as in Section 2.2.6. Set up the equipment as shown in Figure 3-2. Just prior to sampling, leak-check (optional) the train by placing a vacuum gauge at the condenser inlet, pulling a vacuum of at least 250 mm Hg (10 in. Hg), plugging the outlet at the quick disconnect, and then turning off the pump. The vacuum should remain stable for at least 0.5 minute. Evacuate the flexible bag. Connect the probe and place it in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Next, connect the bag and make sure that all connections are tight and leak free.

3.2.3 Sample at a constant rate. The sampling run should be simultaneous with, and for the same total length of time as, the pollutant emission rate determination. Collection of at least 30 liters (1.00 $ft^3$) of sample gas is recommended; however, smaller volumes may be collected, if desired.

3.2.4 Obtain one integrated flue gas sample during each pollutant emission rate determination. Within 8 hours after the sample is taken, analyze it for percent $CO_2$ and percent $O_2$ using either an Orsat analyzer or a Fyrite-type combustion gas analyzer. If an Orsat analyzer is used, it is recommended that the Orsat leak-check described in Section 5 be performed before this determination; however, the check is optional. Determine the percentage of the gas that is $N_2$ and CO by subtracting the sum of the percent $CO_2$ and percent $O_2$

---

[1] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

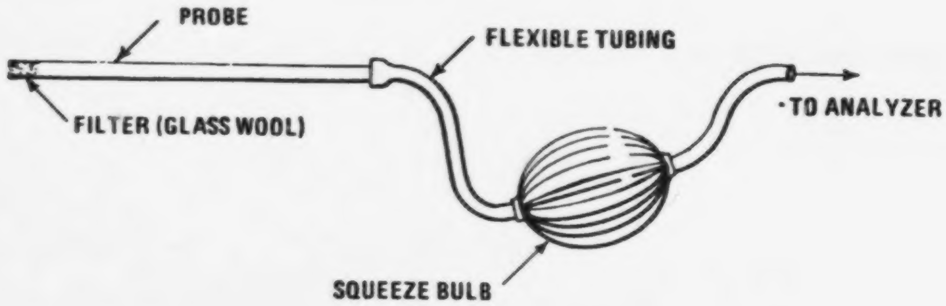

PROBE
FLEXIBLE TUBING
FILTER (GLASS WOOL)
TO ANALYZER
SQUEEZE BULB

Figure 3-1. Grab-sampling train.

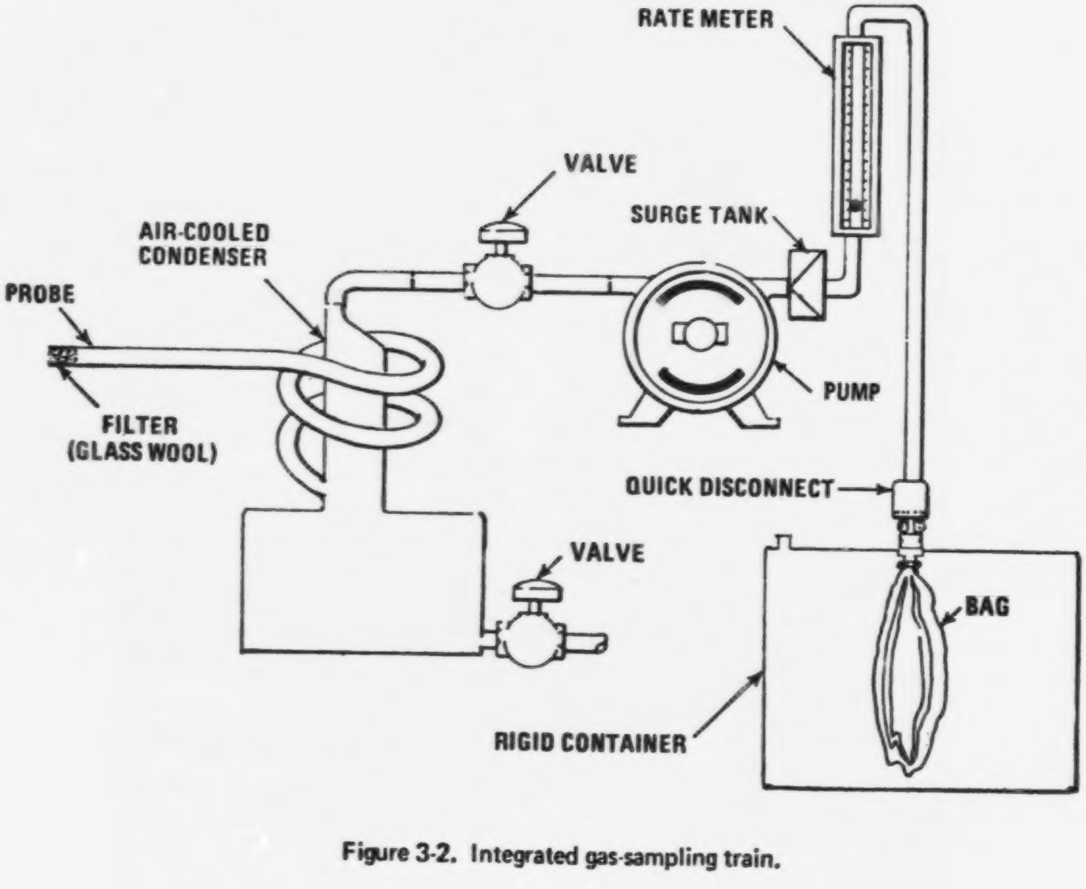

RATE METER
VALVE
SURGE TANK
AIR-COOLED CONDENSER
PROBE
PUMP
FILTER (GLASS WOOL)
QUICK DISCONNECT
VALVE
BAG
RIGID CONTAINER

Figure 3-2. Integrated gas-sampling train.

from 100 percent. Calculate the dry molecular weight as indicated in Section 6.3.

3.2.5 Repeat the analysis and calculation procedures until the individual dry molecular weights for any three analyses differ from their mean by no more than 0.3 g/g-mole (0.3 lb/lb-mole). Average these three molecular weights, and report the results to the nearest 0.1 g/g-mole (0.1 lb/lb-mole).

3.3 Multi-Point, Integrated Sampling and Analytical Procedure.

3.3.1 Unless otherwise specified by the Administrator, a minimum of eight traverse points shall be used for circular stacks having diameters less then 0.61 m (24 in.), a minimum of nine shall be used for rectangular stacks having equivalent diameters less than 0.61 m (24 in.). and a minimum of twelve traverse points shall be used for all other cases. The traverse points shall be located according to Method 1. The use of fewer points is subject to approval of the Administrator.

3.3.2 Follow the procedures outlined in Sections 3.2.2 through 3.2.5, except for the following: traverse all sampling points and sample at each point for an equal length of time. Record sampling data as shown in Figure 3-3.

## 4. *Emission Rate Correction Factor or Excess Air Determination*

NOTE.—A Fyrite-type combustion gas analyzer is not acceptable for excess air or emission rate correction factor determination, unless approved by the Administrator. If both percent $CO_2$ and percent $O_2$ are measured, the analytical results of any of the three procedures given below may also be used for calculating the dry molecular weight.

Each of the three procedures below shall be used only when specified in an applicable subpart of the standards. The use of these procedures for other purposes must have specific prior approval of the Administrator.

4.1 Single-Point, Grab Sampling and Analytical Procedure.

4.1.1 The sampling point in the duct shall either be at the centroid of the cross-section or at a point no closer to the walls than 1.00 m (3.3 ft), unless otherwise specified by the Administrator.

4.1.2 Set up the equipment as shown in Figure 3-1, making sure all connections ahead of the analyzer are tight and leak-free. Leak-check the Orsat analyzer according to the procedure described in Section 5. This leak-check is mandatory.

4.1.3 Place the probe in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Draw a sample into the analyzer. For emission rate correction factor determination, immediately analyze the sample, as outlined in Sections 4.1.4 and 4.1.5, for percent $CO_2$ or percent $O_2$. If excess air is desired, proceed as follows: (1) immediately analyze the sample, as in Sections 4.1.4 and 4.1.5, for percent $CO_2$, $O_2$, and CO; (2) determine the percentage of the gas that is $N_2$ by subtracting the sum of the percent $CO_2$, percent $O_2$, and percent CO from 100 percent; and (3) calculate percent excess air as outlined in Section 6.2.

4.1.4 To ensure complete absorption of the $CO_2$, $O_2$, or if applicable, CO, make repeated passes through each absorbing solution until two consecutive readings are the same. Several passes (three or four) should be made between readings. (If constant readings cannot be obtained after three consecutive readings, replace the absorbing solution.)

4.1.5 After the analysis is completed, leak-check (mandatory) the Orsat analyzer once again, as described in Section 5. For the results of the analysis to be valid, the Orsat analyzer must pass this leak test before and after the analysis. NOTE.—Since this single-point, grab sampling and analytical procedure is normally conducted in conjunction with a single-point, grab sampling and analytical procedure for a pollutant, only one analysis is ordinarily conducted. Therefore, great care must be taken to obtain a valid sample and analysis. Although in most cases only $CO_2$ or $O_2$ is required, it is recommended that both $CO_2$ and $O_2$ be measured, and that Citation 5 in the Bibliography be used to validate the analytical data.

4.2 Single-Point, Integrated Sampling and Analytical Procedure.

4.2.1 The sampling point in the duct shall be located as specified in Section 4.1.1.

4.2.2 Leak-check (mandatory) the flexible bag as in Section 2.2.6. Set up the equipment as shown in Figure 3-2. Just prior to sampling, leak-check (mandatory) the train by placing a vacuum gauge at the condenser inlet, pulling a vacuum of at least 250 mm Hg (10 in. Hg), plugging the outlet at the quick disconnect, and then turning off the pump. The vacuum shall remain stable for at least 0.5 minute. Evacuate the flexible bag. Connect the probe and place it in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Next, connect the bag and make sure that all connections are tight and leak free.

4.2.3 Sample at a constant rate, or as specified by the Administrator. The sampling run must be simultaneous with, and for the same total length of time as, the pollutant emission rate determination. Collect at least 30 liters (1.00 ft³) of sample gas. Smaller volumes may be collected, subject to approval of the Administrator.

4.2.4 Obtain one integrated flue gas sample during each pollutant emission rate determination. For emission rate correction factor determination, analyze the sample within 4 hours after it is taken for percent $CO_2$ or percent $O_2$ (as outlined in Sections 4.2.5 through 4.2.7). The Orsat analyzer must be leak-checked (see Section 5) before the analysis. If excess air is desired, proceed as follows: (1) within 4 hours after the sample is taken, analyze it (as in Sections 4.2.5 through 4.2.7) for percent $CO_2$, $O_2$, and CO; (2) determine the percentage of the gas that is $N_2$ by subtracting the sum of the percent $CO_2$, percent $O_2$, and percent CO from 100 percent; (3) calculate percent excess air, as outlined in Section 6.2.

4.2.5 To ensure complete absorption of the $CO_2$, $O_2$, or if applicable, CO, make repeated passes through each absorbing solution until two consecutive readings are the same. Several passes (three or four) should be made between readings. (If constant readings cannot be obtained after three consecutive readings, replace the absorbing solution.)

4.2.6 Repeat the analysis until the following criteria are met:

4.2.6.1 For percent $CO_2$, repeat the analytical procedure until the results of any three analyses differ by no more than (a) 0.3 percent by volume when $CO_2$ is greater than 4.0 percent or (b) 0.2 percent by volume when $CO_2$ is less than or equal to 4.0 percent. Average the three acceptable values of percent $CO_2$ and report the results to the nearest 0.1 percent.

4.2.6.2 For percent $O_2$, repeat the analytical procedure until the results of any three analyses differ by no more than (a) 0.3 percent by volume when $O_2$ is less than 15.0 percent or (b) 0.2 percent by volume when $O_2$ is greater than 15.0 percent. Average the three acceptable values of percent $O_2$ and report the results to the nearest 0.1 percent.

4.2.6.3 For percent CO, repeat the analytical procedure until the results of any three analyses differ by no more than 0.3 percent. Average the three acceptable values of percent CO and report the results to the nearest 0.1 percent.

4.2.7 After the analysis is completed, leak-check (mandatory) the Orsat analyzer once again, as described in Section 5. For the results of the analysis to be valid, the Orsat analyzer must pass this leak test before and after the analysis. Note: Although in most instances only $CO_2$ or $O_2$ is required, it is recommended that both $CO_2$ and $O_2$ be measured, and that Citation 5 in the Bibliography be used to validate the analytical data.

4.3 Multi-Point, Integrated Sampling and Analytical Procedure.

4.3.1 Both the minimum number of sampling points and the sampling point location shall be as specified in Section 3.3.1 of this method. The use of fewer points than specified is subject to the approval of the Administrator.

4.3.2 Follow the procedures outlined in Sections 4.2.2 through 4.2.7, except for the following: Traverse all sampling points and sample at each point for an equal length of time. Record sampling data as shown in Figure 3-3.

## 5. *Leak-Check Procedure for Orsat Analyzers*

Moving an Orsat analyzer frequently causes it to leak. Therefore, an Orsat analyzer should be thoroughly leak-checked on site before the flue gas sample is introduced into it. The procedure for leak-checking an Orsat analyzer is:

5.1.1 Bring the liquid level in each pipette up to the reference mark on the capillary tubing and then close the pipette stopcock.

5.1.2 Raise the leveling bulb sufficiently to bring the confining liquid meniscus onto the graduated portion of the burette and then close the manifold stopcock.

5.1.3 Record the meniscus position.

5.1.4 Observe the meniscus in the burette and the liquid level in the pipette for movement over the next 4 minutes.

5.1.5 For the Orsat analyzer to pass the leak-check, two conditions must be met.

5.1.5.1 The liquid level in each pipette must not fall below the bottom of the capillary tubing during this 4-minute interval.

5.1.5.2 The meniscus in the burette must not change by more than 0.2 ml during this 4-minute interval.

5.1.6 If the analyzer fails the leak-check procedure, all rubber connections and stopcocks should be checked until the cause of the leak is identified. Leaking stopcocks must be disassembled, cleaned, and regreased. Leaking rubber connections must be replaced. After the analyzer is reassembled, the leak-check procedure must be repeated.

## 6. *Calculations*

6.1 Nomenclature.

$M_d$ = Dry molecular weight, g/g-mole (lb/lb-mole).
%EA = Percent excess air.
$\%CO_2$ = Percent $CO_2$ by volume (dry basis).
$\%O_2$ = Percent $O_2$ by volume (dry basis).
%CO = Percent CO by volume (dry basis).
$\%N_2$ = Percent $N_2$ by volume (dry basis).
0.264 = Ratio of $O_2$ to $N_2$ in air, v/v.
0.280 = Molecular weight of $N_2$ or CO, divided by 100.
0.320 = Molecular weight of $O_2$ divided by 100.
0.440 = Molecular weight of $CO_2$ divided by 100.

6.2 Percent Excess Air. Calculate the percent excess air (if applicable), by substituting the appropriate values of percent $O_2$, CO, and $N_2$ (obtained from Section 4.1.3 or 4.2.4) into Equation 3-1.

$$\%EA=\left[\frac{\%O_2-0.5\%CO}{0.264\,\%N_2(\%O_2-0.5\,\%CO)}\right]100$$

Equation 3-1

NOTE.—The equation above assumes that ambient air is used as the source of $O_2$ and that the fuel does not contain appreciable amounts of $N_2$ (as do coke oven or blast furnace gases). For those cases when appreciable amounts of $N_2$ are present (coal, oil, and natural gas do not contain appreciable amounts of $N_2$) or when oxygen enrichment is used, alternate methods, subject to approval of the Administrator, are required.

6.3 Dry Molecular Weight. Use Equation 3-2 to calculate the dry molecular weight of the stack gas

$$M_d=0.440(\%CO_2)+0.320(\%O_2)+0.280(\%N_2+\%CO)$$

Equation 3-2

NOTE.—The above equation does not consider argon in air (about 0.9 percent, molecular weight of 37.7). A negative error of about 0.4 percent is introduced. The tester may opt to include argon in the analysis using procedures subject to approval of the Administrator.

7. *Bibliography*

1. Altshuller, A. P. Storage of Gases and Vapors in Plastic Bags. International Journal of Air and Water Pollution. *6*:75-81. 1963.

2. Conner, William D. and J. S. Nader. Air Sampling Plastic Bags. Journal of the American Industrial Hygiene Association. *25*:291-297. 1964.

3. Burrell Manual for Gas Analysts, Seventh edition. Burrell Corporation, 2223 Fifth Avenue, Pittsburgh, Pa. 15219. 1951.

4. Mitchell, W. J. and M. R. Midgett. Field Reliability of the Orsat Analyzer. Journal of Air Pollution Control Association *26*:491-495. May 1976.

5. Shigehara, R. T., R. M. Neulicht, and W. S. Smith. Validating Orsat Analysis Data from Fossil Fuel-Fired Units. Stack Sampling News. *4*(2):21-26. August, 1976.

| TIME | TRAVERSE PT. | Q 1pm | % DEV.[a] |
|---|---|---|---|
| | | | |
| | | | |
| | | | |
| | | | |
| | AVERAGE | | |

[a] $\% \text{ DEV} = \left(\frac{Q - Q_{avg}}{Q_{avg}}\right) 100$ (MUST BE ≤10%)

Figure 3.3. Sampling rate data.

# METHOD 4

# DETERMINATION OF MOISTURE IN STACK GASES

## 1. *Principle and Applicability*

**1.1 Principle.** A gas sample is extracted at a constant rate from the source; moisture is removed from the sample stream and determined either volumetrically or gravimetrically.

**1.2 Applicability.** This method is applicable for determining the moisture content of stack gas.

Two procedures are given. The first is a reference method, for accurate determinations of moisture content (such as are needed to calculate emission data). The second is an approximation method, which provides estimates of percent moisture to aid in setting isokinetic sampling rates prior to a pollutant emission measurement run. The approximation method described herein is only a suggested approach; alternative means for approximating the moisture content, e.g., drying tubes, wet bulb-dry bulb techniques, condensation techniques, stoichiometric calculations, previous experience, etc., are also acceptable.

The reference method is often conducted simultaneously with a pollutant emission measurement run; when it is, calculation of percent isokinetic, pollutant emission rate, etc., for the run shall be based upon the results of the reference method or its equivalent; these calculations shall not be based upon the results of the approximation method, unless the approximation method is shown, to the satisfaction of the Administrator, U.S. Environmental Protection Agency, to be capable of yielding results within 1 percent $H_2O$ of the reference method.

NOTE.—The reference method may yield questionable results when applied to saturated gas streams or to streams that contain water droplets. Therefore, when these conditions exist or are suspected, a second determination of the moisture content shall be made simultaneously with the reference method, as follows: Assume that the gas stream is saturated. Attach a temperature sensor [capable of measuring to ±1° C (2° F)] to the reference method probe. Measure the stack gas temperature at each traverse point (see Section 2.2.1) during the reference method traverse; calculate the average stack gas temperature. Next, determine the moisture percentage, either by: (1) using a psychrometric chart and making appropriate corrections if stack pressure is different from that of the chart, or (2) using saturation vapor pressure tables. In cases where the psychrometric chart or the saturation vapor pressure tables are not applicable (based on evaluation of the process), alternate methods, subject to the approval of the Administrator, shall be used.

## 2. *Reference Method*

The procedure described in Method 5 for determining moisture content is acceptable as a reference method.

**2.1 Apparatus.** A schematic of the sampling train used in this reference method is shown in Figure 4-1. All components shall be maintained and calibrated according to the procedure outlined in Method 5.

**2.1.1 Probe.** The probe is constructed of stainless steel or glass tubing, sufficiently heated to prevent water condensation, and is equipped with a filter, either in-stack (e.g., a plug of glass wool inserted into the end of the probe) or heated out-stack (e.g., as described in Method 5), to remove particulate matter.

When stack conditions permit, other metals or plastic tubing may be used for the probe, subject to the approval of the Administrator.

**2.1.2 Condenser.** The condenser consists of four impingers connected in series with ground glass, leak-free fittings or any similarly leak-free non-contaminating fittings. The first, third, and fourth impingers shall be of the Greenburg-Smith design, modified by replacing the tip with a 1.3 centimeter (½ inch) ID glass tube extending to about 1.3 cm (½ in.) from the bottom of the flask. The second impinger shall be of the Greenburg-Smith design with the standard tip. Modifications (e.g., using flexible connections between the impingers, using materials other than glass, or using flexible vacuum lines to connect the filter holder to the condenser) may be used, subject to the approval of the Administrator.

The first two impingers shall contain known volumes of water, the third shall be empty, and the fourth shall contain a known weight of 6- to 16-mesh indicating type silica gel, or equivalent desiccant. If the silica gel has been previously used, dry at 175° C (350° F) for 2 hours. New silica gel may be used as received. A thermometer, capable of measuring temperature to within 1° C (2° F), shall be placed at the outlet of the fourth impinger, for monitoring purposes.

Alternatively, any system may be used (subject to the approval of the Administrator) that cools the sample gas stream and allows measurement of both the water that has been condensed and the moisture leaving the condenser, each to within 1 ml or 1 g. Acceptable means are to measure the condensed water, either gravimetrically or volumetrically, and to measure the moisture leaving the condenser by: (1) monitoring the temperature and pressure at the exit of the condenser and using Dalton's law of partial pressures, or (2) passing the sample gas stream through a tared silica gel (or equivalent desiccant) trap, with exit gases kept below 20° C (68° F), and determining the weight gain.

If means other than silica gel are used to determine the amount of moisture leaving the condenser, it is recommended that silica gel (or equivalent) still be used between the condenser system and pump, to prevent moisture condensation in the pump and metering devices and to avoid the need to make corrections for moisture in the metered volume.

**2.1.3 Cooling System.** An ice bath container and crushed ice (or equivalent) are used to aid in condensing moisture.

**2.1.4 Metering System.** This system includes a vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3° C (5.4° F), dry gas meter capable of measuring volume to within 2 percent, and related equipment as shown in Figure 4-1. Other metering systems, capable of maintaining a constant sampling rate and determining sample gas volume, may be used, subject to the approval of the Administrator.

**2.1.5 Barometer.** Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg) may be used. In many cases, the barometric reading may be obtained from a nearby national weather service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and the sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

**2.1.6 Graduated Cylinder and/or Balance.** These items are used to measure condensed water and moisture caught in the silica gel to within 1 ml or 0.5 g. Graduated cylinders shall have subdivisions no greater than 2 ml. Most laboratory balances are capable of weighing to the nearest 0.5 g or less. These balances are suitable for use here.

**2.2 Procedure.** The following procedure is written for a condenser system (such as the impinger system described in Section 2.1.2) incorporating volumetric analysis to measure the condensed moisture, and silica gel and gravimetric analysis to measure the moisture leaving the condenser.

2.2.1 Unless otherwise specified by the Administrator, a minimum of eight traverse points shall be used for circular stacks having diameters less than 0.61 m (24 in.), a minimum of nine points shall be used for rectangular stacks having equivalent diameters less than 0.61 m (24 in.), and a minimum of twelve traverse points shall be used in all other cases. The traverse points shall be located according to Method 1. The use of fewer points is subject to the approval of the Administrator. Select a suitable probe and probe length such that all traverse points can be sampled. Consider sampling from opposite sides of the stack (four total sampling ports) for large stacks, to permit use of shorter probe lengths. Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point. Place known volumes of water in the first two impingers. Weigh and record the weight of the silica gel to the nearest 0.5 g, and transfer the silica gel to the fourth impinger; alternatively, the silica gel may first be transferred to the impinger, and the weight of the silica gel plus impinger recorded.

2.2.2 Select a total sampling time such that a minimum total gas volume of 0.60 scm (21 scf) will be collected, at a rate no greater than 0.021 $m^3$/min (0.75 cfm). When both moisture content and pollutant emission rate

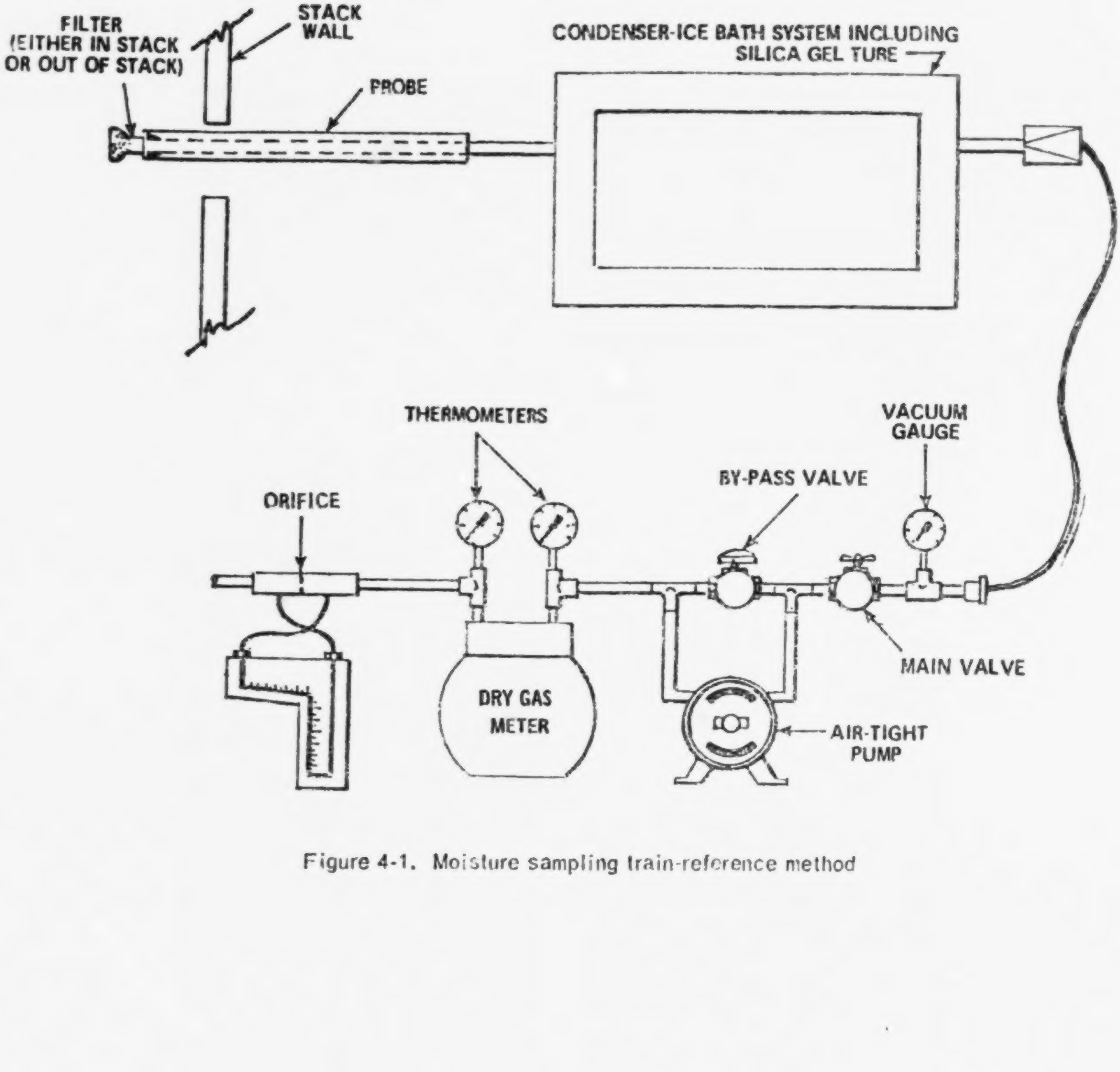

FILTER
(EITHER IN STACK
OR OUT OF STACK)
STACK
WALL
PROBE
CONDENSER-ICE BATH SYSTEM INCLUDING
SILICA GEL TUBE
THERMOMETERS
VACUUM
GAUGE
BY-PASS VALVE
ORIFICE
MAIN VALVE
DRY GAS
METER
AIR-TIGHT
PUMP

Figure 4-1. Moisture sampling train-reference method

PLANT____________________

LOCATION____________________

OPERATOR____________________

DATE____________________

RUN NO.____________________

AMBIENT TEMPERATURE____________________

BAROMETRIC PRESSURE____________________

PROBE LENGTH m(ft)____________________

SCHEMATIC OF STACK CROSS SECTION

| TRAVERSE POINT NUMBER | SAMPLING TIME (θ), min. | STACK TEMPERATURE °C (°F) | PRESSURE DIFFERENTIAL ACROSS ORIFICE METER (ΔH), mm(in.) $H_2O$ | METER READING GAS SAMPLE VOLUME $m^3$ ($ft^3$) | $\Delta V_m$ $m^3$ ($ft^3$) | GAS SAMPLE TEMPERATURE AT DRY GAS METER INLET ($T_{m_{in}}$), °C (°F) | OUTLET ($T_{m_{out}}$), °C (°F) | TEMPERATURE OF GAS LEAVING CONDENSER OR LAST IMPINGER, °C (°F) |
|---|---|---|---|---|---|---|---|---|
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| TOTAL | | | | | | Avg. | Avg. | |
| AVERAGE | | | | | | Avg.. | | |

Figure 4-2. Field moisture determination-reference method.

are to be determined, the moisture determination shall be simultaneous with, and for the same total length of time as, the pollutant emission rate run, unless otherwise specified in an applicable subpart of the standards.

2.2.3 Set up the sampling train as shown in Figure 4-1. Turn on the probe heater and (if applicable) the filter heating system to temperatures of about 120° C (248° F), to prevent water condensation ahead of the condenser; allow time for the temperatures to stabilize. Place crushed ice in the ice bath container. It is recommended, but not required, that a leak check be done, as follows: Disconnect the probe from the first impinger or (if applicable) from the filter holder. Plug the inlet to the first impinger (or filter holder) and pull a 380 mm (15 in.) Hg vacuum; a lower vacuum may be used, provided that it is not exceeded during the test. A leakage rate in excess of 4 percent of the average sampling rate or 0.00057 m³/min (0.02 cfm), whichever is less, is unacceptable. Following the leak check, reconnect the probe to the sampling train.

2.2.4 During the sampling run, maintain a sampling rate within 10 percent of constant rate, or as specified by the Administrator. For each run, record the data required on the example data sheet shown in Figure 4-2. Be sure to record the dry gas meter reading at the beginning and end of each sampling time increment and whenever sampling is halted. Take other appropriate readings at each sample point, at least once during each time increment.

2.2.5 To begin sampling, position the probe tip at the first traverse point. Immediately start the pump and adjust the flow to the desired rate. Traverse the cross section, sampling at each traverse point for an equal length of time. Add more ice and, if necessary, salt to maintain a temperature of less than 20° C (68° F) at the silica gel outlet.

2.2.6 After collecting the sample, disconnect the probe from the filter holder (or from the first impinger) and conduct a leak check (mandatory) as described in Section 2.2.3. Record the leak rate. If the leakage rate exceeds the allowable rate, the tester shall either reject the test results or shall correct the sample volume as in Section 6.3 of Method 5. Next, measure the volume of the moisture condensed to the nearest ml. Determine the increase in weight of the silica gel (or silica gel plus impinger) to the nearest 0.5 g. Record this information (see example data sheet, Figure 4-3) and calculate the moisture percentage, as described in 2.3 below.

2.3 Calculations. Carry out the following calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

| | IMPINGER VOLUME, ml | SILICA GEL WEIGHT, g |
|---|---|---|
| FINAL | | |
| INITIAL | | |
| DIFFERENCE | | |

Figure 4-3. Analytical data—reference method.

2.3.1 Nomenclature.

$B_{ws}$ = Proportion of water vapor, by volume, in the gas stream.

$M_w$ = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole).

$P_m$ = Absolute pressure (for this method, same as barometric pressure) at the dry gas meter, mm Hg (in. Hg).

$P_{std}$ = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

$R$ = Ideal gas constant, 0.06236 (mm Hg) (m³)/(g-mole) (°K) for metric units and 21.85 (in. Hg) (ft³)/(lb-mole) (°R) for English units.

$T_m$ = Absolute temperature at meter, °K (°R).

$T_{std}$ = Standard absolute temperature, 293° K (528° R).

$V_m$ = Dry gas volume measured by dry gas meter, dcm (dcf).

$\Delta V_m$ = Incremental dry gas volume measured by dry gas meter at each traverse point, dcm (dcf).

$V_{m(std)}$ = Dry gas volume measured by the dry gas meter, corrected to standard conditions, dscm (dscf).

$V_{wc(std)}$ = Volume of water vapor condensed corrected to standard conditions, scm (scf).

$V_{wsg(std)}$ = Volume of water vapor collected in silica gel corrected to standard conditions, scm (scf).

$V_f$ = Final volume of condenser water, ml.

$V_i$ = Initial volume, if any, of condenser water, ml.

$W_f$ = Final weight of silica gel or silica gel plus impinger, g.

$W_i$ = Initial weight of silica gel or silica gel plus impinger, g.

$Y$ = Dry gas meter calibration factor.

$\rho_w$ = Density of water, 0.9982 g/ml (0.002201 lb/ml).

2.3.2 Volume of water vapor condensed.

$$V_{wc(std)} = \frac{(V_f - V_i)\rho_w R T_{std}}{P_{std} M_w} = K_1 (V_f - V_i)$$

Equation 4-1

where:

$K_1$ = 0.001333 m³/ml for metric units
= 0.04707 ft³/ml for English units.

2.3.3 Volume of water vapor collected in silica gel.

$$V_{wsg(std)} = \frac{(W_f - W_i) R T_{std}}{P_{std} M_w} = K_2 (W_f - W_i)$$

Equation 4-2

where:

$K_2$ = 0.001335 m³/g for metric units
= 0.04715 ft³/g for English units.

2.3.4 Sample gas volume.

$$V_{m(std)} = V_m Y \left(\frac{P_m}{P_{std}}\right)\left(\frac{T_{std}}{T_m}\right) = K_3 Y \frac{V_m P_m}{T_m}$$

Equation 4-3

where:

$K_3$ = 0.3858 °K/mm Hg for metric units
= 17.64 °R/in. Hg for English units.

NOTE.—If the post-test leak rate (Section 2.2.6) exceeds the allowable rate, correct the value of $V_m$ in Equation 4-3, as described in Section 6.3 of Method 5.

2.3.5 Moisture Content.

$$B_{ws} = \frac{V_{wc(std)} + V_{wsg(std)}}{V_{wc(std)} + V_{wsg(std)} + V_{m(std)}}$$

Equation 4-4

NOTE.—In saturated or moisture droplet-laden gas streams, two calculations of the moisture content of the stack gas shall be made, one using a value based upon the saturated conditions (see Section 1.2), and another based upon the results of the impinger analysis. The lower of these two values of $B_{ws}$ shall be considered correct.

2.3.6 Verification of constant sampling rate. For each time increment, determine the $\Delta V_m$. Calculate the average. If the value for any time increment differs from the average by more than 10 percent, reject the results and repeat the run.

## 3. *Approximation Method*

The approximation method described below is presented only as a suggested method (see Section 1.2).

3.1 Apparatus.

3.1.1 Probe. Stainless steel or glass tubing, sufficiently heated to prevent water condensation and equipped with a filter (either in-stack or heated out-stack) to remove particulate matter. A plug of glass wool, inserted into the end of the probe, is a satisfactory filter.

3.1.2 Impingers. Two midget impingers, each with 30 ml capacity, or equivalent.

3.1.3 Ice Bath. Container and ice, to aid in condensing moisture in impingers.

3.1.4 Drying Tube. Tube packed with new or regenerated 6- to 16-mesh indicating-type silica gel (or equivalent desiccant), to dry the sample gas and to protect the meter and pump.

3.1.5 Valve. Needle valve, to regulate the sample gas flow rate.

3.1.6 Pump. Leak-free, diaphragm type, or equivalent, to pull the gas sample through the train.

3.1.7 Volume meter. Dry gas meter, sufficiently accurate to measure the sample volume within 2%, and calibrated over the range of flow rates and conditions actually encountered during sampling.

3.1.8 Rate Meter. Rotameter, to measure the flow range from 0 to 3 lpm (0 to 0.11 cfm).

3.1.9 Graduated Cylinder. 25 ml.

3.1.10 Barometer. Mercury, aneroid, or other barometer, as described in Section 2.1.5 above.

3.1.11 Vacuum Gauge. At least 760 mm Hg (30 in. Hg) gauge, to be used for the sampling leak check.

3.2 Procedure.

3.2.1 Place exactly 5 ml distilled water in each impinger. Assemble the apparatus without the probe as shown in Figure 4-4. Leak check the train by placing a vacuum gauge at the inlet to the first impinger and drawing a vacuum of at least 250 mm Hg (10 in. Hg), plugging the outlet of the rotameter, and then turning off the pump. The vacuum shall remain constant for at least one minute. Carefully release the vacuum gauge before unplugging the rotameter end.

3.2.2 Connect the probe, insert it into the stack, and sample at a constant rate of 2 lpm (0.071 cfm). Continue sampling until the dry gas meter registers about 30 liters (1.1 ft³) or until visible liquid droplets are carried over from the first impinger to the second. Record temperature, pressure, and dry gas meter readings as required by Figure 4-5.

3.2.3 After collecting the sample, combine the contents of the two impingers and measure the volume to the nearest 0.5 ml.

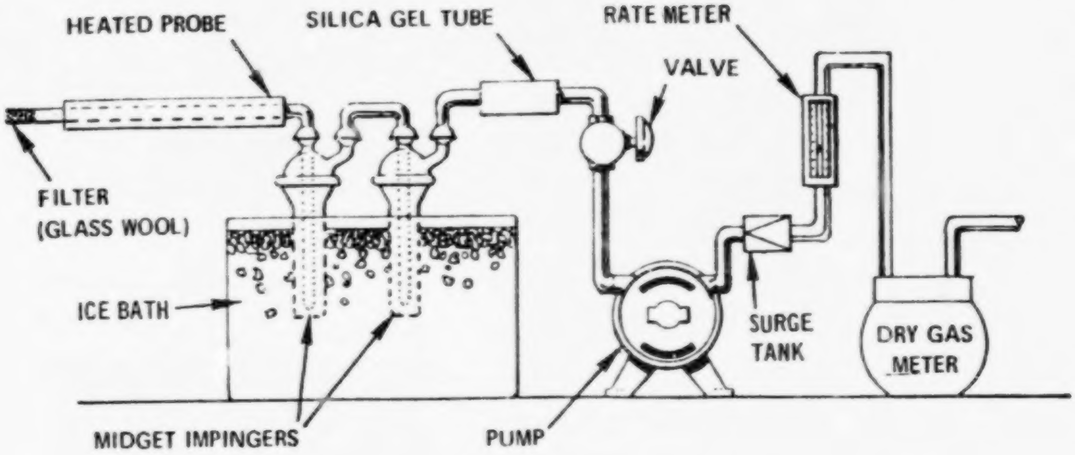

HEATED PROBE
SILICA GEL TUBE
RATE METER
VALVE
FILTER (GLASS WOOL)
ICE BATH
SURGE TANK
DRY GAS METER
MIDGET IMPINGERS
PUMP

Figure 4-4. Moisture-sampling train - approximation method.

LOCATION ____________________ COMMENTS

TEST ____________________

DATE ____________________

OPERATOR ____________________

BAROMETRIC PRESSURE ____________________

| CLOCK TIME | GAS VOLUME THROUGH METER, (Vm), $m^3$ ($ft^3$) | RATE METER SETTING $m^3$/min. ($ft^3$/min.) | METER TEMPERATURE, °C (°F) |
|---|---|---|---|
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |

Figure 4-5. Field moisture determination - approximation method.

3.3 Calculations. The calculation method presented is designed to estimate the moisture in the stack gas; therefore, other data, which are only necessary for accurate moisture determinations, are not collected. The following equations adequately estimate the moisture content, for the purpose of determining isokinetic sampling rate settings.

3.3.1 Nomenclature.

$B_{wm}$ = Approximate proportion, by volume, of water vapor in the gas stream leaving the second impinger, 0.025.
$B_{ws}$ = Water vapor in the gas stream, proportion by volume.
$M_w$ = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole)
$P_m$ = Absolute pressure (for this method, same as barometric pressure) at the dry gas meter.
$P_{std}$ = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).
$R$ = Ideal gas constant, 0.06236 (mm Hg) ($m^3$)/(g-mole) (°K) for metric units and 21.85 (in. Hg) ($ft^3$)/lb-mole) (°R) for English units.
$T_m$ = Absolute temperature at meter, °K (°R)
$T_{std}$ = Standard absolute temperature, 293° K (528° R)
$V_f$ = Final volume of impinger contents, ml.
$V_i$ = Initial volume of impinger contents, ml.
$V_m$ = Dry gas volume measured by dry gas meter, dcm (dcf).
$V_{m(std)}$ = Dry gas volume measured by dry gas meter, corrected to standard conditions, dscm (dscf).
$V_{wc(std)}$ = Volume of water vapor condensed, corrected to standard conditions, scm (scf).
$\rho_w$ = Density of water, 0.9982 g/ml (0.002201 lb/ml).

3.3.2 Volume of water vapor collected.

$$V_{wc} = \frac{(V_f - V_i)\rho_w R T_{std}}{P_{std} M_w} = K_1 (V_f - V_i)$$

Equation 4-5

where:
$K_1$ = 0.001333 $m^3$/ml for metric units
= 0.04707 $ft^3$/ml for English units.

3.3.3 Gas volume.

$$V_{m(std)} = V_m \left(\frac{P_m}{P_{std}}\right)\left(\frac{T_{std}}{T_m}\right) = K_2 \frac{V_m P_m}{T_m}$$

Equation 4-6

where:
$K_2$ = 0.3858 °K/mm Hg for metric units
= 17.64 °R/in. Hg for English units

3.3.4 Approximate moisture content.

$$B_{ws} = \frac{V_{wc}}{V_{wc} + V_{m(std)}} + B_{wm} = \frac{V_{wc}}{V_{wc} + V_{m(std)}} + (0.025)$$

Equation 4-7

## 4. *Calibration*

4.1 For the reference method, calibrate equipment as specified in the following sections of Method 5: Section 5.3 (metering system); Section 5.5 (temperature gauges); and Section 5.7 (barometer). The recommended leak check of the metering system (Section 5.6 of Method 5) also applies to the reference method. For the approximation method, use the procedures outlined in Section 5.1.1 of Method 6 to calibrate the metering system, and the procedure of Method 5, Section 5.7 to calibrate the barometer.

## 5. *Bibliography*

1. Air Pollution Engineering Manual (Second Edition). Danielson, J. A. (ed.). U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards. Research Triangle Park, N.C. Publication No. AP-40. 1973.

2. Devorkin, Howard, et al. Air Pollution Source Testing Manual. Air Pollution Control District, Los Angeles, Calif. November, 1963.

3. Methods for Determination of Velocity, Volume, Dust and Mist Content of Gases. Western Precipitation Division of Joy Manufacturing Co., Los Angeles, Calif. Bulletin WP-50. 1968.

# METHOD 5

## DETERMINATION OF PARTICULATE EMISSIONS FROM STATIONARY SOURCES

### 1. *Principle and Applicability*

1.1 Principle. Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at a temperature in the range of 120±14° C (248±25° F) or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator, U.S. Environmental Protection Agency, for a particular application. The particulate mass, which includes any material that condenses at or above the filtration temperature, is determined gravimetrically after removal of uncombined water.

1.2 Applicability. This method is applicable for the determination of particulate emissions from stationary sources.

### 2. *Apparatus*

2.1 Sampling Train. A schematic of the sampling train used in this method is shown in Figure 5-1. Complete construction details are given in APTD-0581 (Citation 2 in Section 7); commercial models of this train are also available. For changes from APTD-0581 and for allowable modifications of the train shown in Figure 5-1, see the following subsections.

The operating and maintenance procedures for the sampling train are described in APTD-0576 (Citation 3 in Section 7). Since correct usage is important in obtaining valid results, all users should read APTD-0576 and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein. The sampling train consists of the following components:

2.1.1 Probe Nozzle. Stainless steel (316) or glass with sharp, tapered leading edge. The angle of taper shall be ≤30° and the taper shall be on the outside to preserve a constant internal diameter. The proble nozzle shall be of the button-hook or elbow design, unless otherwise specified by the Administrator. If made of stainless steel, the nozzle shall be constructed from seamless tubing; other materials of construction may be used, subject to the approval of the Administrator.

A range of nozzle sizes suitable for isokinetic sampling should be available, e.g., 0.32 to 1.27 cm (⅛ to ½ in.)—or larger if higher volume sampling trains are used—inside diameter (ID) nozzles in increments of 0.16 cm (1/16 in.). Each nozzle shall be calibrated according to the procedures outlined in Section 5.

2.1.2 Probe Liner. Borosilicate or quartz glass tubing with a heating system capable of maintaining a gas temperature at the exit end during sampling of 120±14° C (248±25° F), or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator for a particular application. (The tester may opt to operate the equipment at a temperature lower than that specified.) Since the actual temperature at the outlet of the probe is not usually monitored during sampling, probes constructed according to APTD-0581 and utilizing the calibration curves of APTD-0576 (or calibrated according to the procedure outlined in APTD-0576) will be considered acceptable.

Either borosilicate or quartz glass probe liners may be used for stack temperatures up to about 480° C (900° F); quartz liners shall be used for temperatures between 480 and 900° C (900 and 1,650° F). Both types of liners may be used at higher temperatures than specified for short periods of time, subject to the approval of the Administrator. The softening temperature for borosilicate is 830° C (1,508° F), and for quartz it is 1,500° C (2,732° F).

Whenever practical, every effort should be made to use borosilicate or quartz glass probe liners. Alternatively, metal liners (e.g., 316 stainless steel, Incoloy 825,[2] or other corrosion resistant metals) made of seamless tubing may be used, subject to the approval of the Administrator.

2.1.3 Pitot Tube. Type S, as described in Section 2.1 of Method 2, or other device approved by the Administrator. The pitot tube shall be attached to the probe (as shown in Figure 5-1) to allow constant monitoring of the stack gas velocity. The impact (high pressure) opening plane of the pitot tube shall be even with or above the nozzle entry plane (see Method 2, Figure 2-6b) during sampling. The Type S pitot tube assembly shall have a known coefficient, determined as outlined in Section 4 of Method 2.

2.1.4 Differential Pressure Gauge. Inclined manometer or equivalent device (two), as described in Section 2.2 of Method 2. One manometer shall be used for velocity head ($\Delta p$) readings, and the other, for orifice differential pressure readings.

2.1.5 Filter Holder. Borosilicate glass, with a glass frit filter support and a silicone rubber gasket. Other materials of construction (e.g., stainless steel, Teflon, Viton) may be used, subject to approval of the Administrator. The holder design shall provide a positive seal against leakage from the outside or around the filter. The holder shall be attached immediately at the outlet of the probe (or cyclone, if used).

2.1.6 Filter Heating System. Any heating system capable of maintaining a temperature around the filter holder during sampling of 120±14° C (248±25° F), or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator for a particular application. Alternatively, the tester may opt to operate the equipment at a temperature lower than that specified. A temperature gauge capable of measuring temperature to within 3° C (5.4° F) shall be installed so that the temperature around the filter holder can be regulated and monitored during sampling. Heating systems other than the one shown in APTD-0581 may be used.

2.1.7 Condenser. The following system shall be used to determine the stack gas moisture content: Four impingers connected in series with leak-free ground glass fittings or any similar leak-free non-contaminating fittings. The first, third, and fourth impingers shall be of the Greenburg-Smith design, modified by replacing the tip with 1.3 cm (½ in.) ID glass tube extending to about 1.3 cm (½ in.) from the bottom of the flask. The second impinger shall be of the Greenburg-Smith design with the standard tip. Modifications (e.g., using flexible connections between the impingers, using materials other than glass, or using flexible vacuum lines to connect the filter holder to the condenser) may be used, subject to the approval of the Administrator. The first and second impingers shall contain known quantities of water (Section 4.1.3), the third shall be empty, and the fourth shall contain a known weight of silica gel, or equivalent desiccant. A thermometer, capable of measuring temperature to within 1° C (2° F) shall be placed at the outlet of the fourth impinger for monitoring purposes.

Alternatively, any system that cools the sample gas stream and allows measurement of the water condensed and moisture leaving the condenser, each to within 1 ml or 1 g may be used, subject to the approval of the Administrator. Acceptable means are to measure the condensed water either gravimetrically or volumetrically and to measure the moisture leaving the condenser by: (1) monitoring the temperature and pressure at the exit of the condenser and using Dalton's law of partial pressures; or (2) passing the sample gas stream through a tared silica gel (or equivalent desiccant) trap with exit gases kept below 20° C (68° F) and determining the weight gain.

If means other than silica gel are used to determine the amount of moisture leaving the condenser, it is recommended that silica gel (or equivalent) still be used between the condenser system and pump to prevent moisture condensation in the pump and metering devices and to avoid the need to make corrections for moisture in the metered volume.

NOTE.—If a determination of the particulate matter collected in the impingers is desired in addition to moisture content, the impinger system described above shall be used, without modification. Individual States or control agencies requiring this information shall be contacted as to the sample recovery and analysis of the impinger contents.

2.1.8 Metering System. Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3° C (5.4° F), dry gas meter capable of measuring volume to within 2 percent, and related equipment, as shown in Figure 5-1. Other metering systems capable of maintaining sampling rates within 10 percent of isokinetic and of determining sample volumes to within 2

---

[2] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

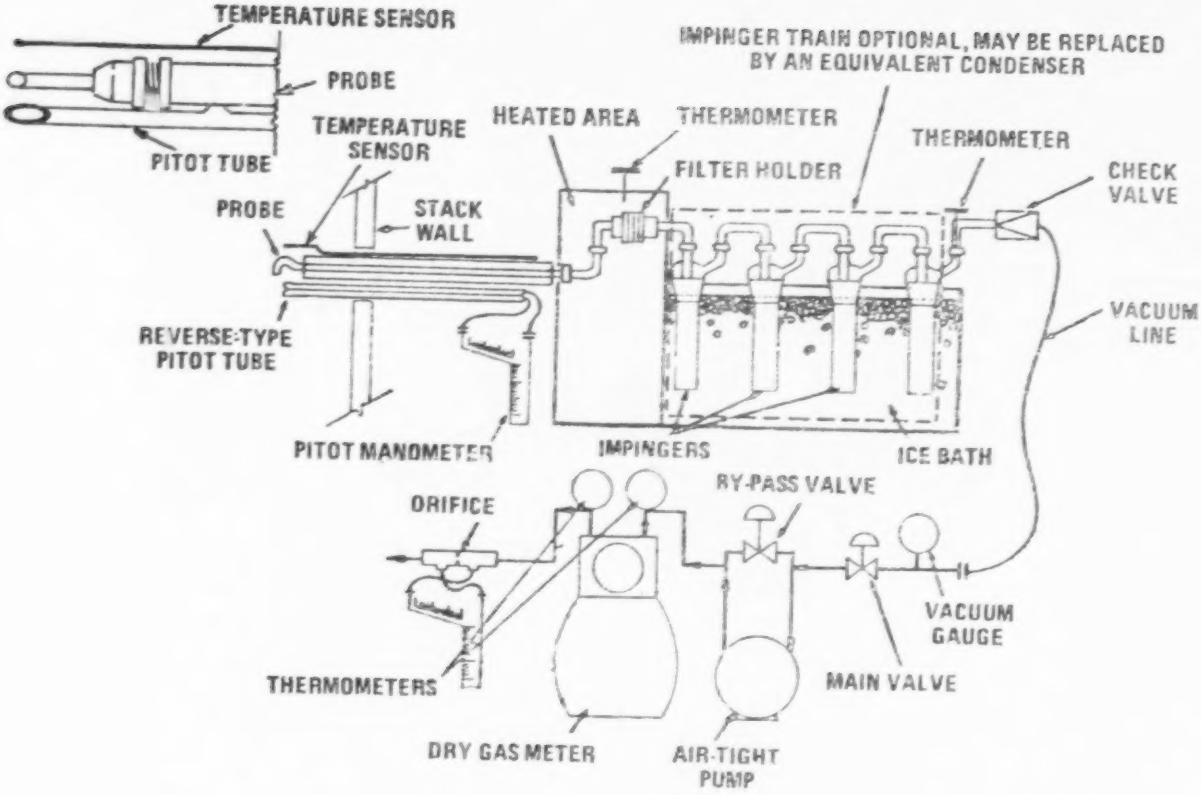

TEMPERATURE SENSOR
PROBE
PITOT TUBE
TEMPERATURE SENSOR
PROBE
STACK WALL
REVERSE-TYPE PITOT TUBE
PITOT MANOMETER
HEATED AREA
THERMOMETER
FILTER HOLDER
IMPINGER TRAIN OPTIONAL, MAY BE REPLACED BY AN EQUIVALENT CONDENSER
THERMOMETER
CHECK VALVE
VACUUM LINE
IMPINGERS
ICE BATH
BY-PASS VALVE
ORIFICE
VACUUM GAUGE
MAIN VALVE
THERMOMETERS
DRY GAS METER
AIR-TIGHT PUMP

Figure 5-1. Particulate-sampling train.

percent may be used, subject to the approval of the Administrator. When the metering system is used in conjunction with a pitot tube, the system shall enable checks of isokinetic rates.

Sampling trains utilizing metering systems designed for higher flow rates than that described in APTD-0581 or APTD-0576 may be used provided that the specifications of this method are met.

2.1.9 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby national weather service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

2.1.10 Gas Density Determination Equipment. Temperature sensor and pressure gauge, as described in Sections 2.3 and 2.4 of Method 2, and gas analyzer, if necessary, as described in Method 3. The temperature sensor shall, preferably, be permanently attached to the pitot tube or sampling probe in a fixed configuration, such that the tip of the sensor extends beyond the leading edge of the probe sheath and does not touch any metal. Alternatively, the sensor may be attached just prior to use in the field. Note, however, that if the temperature sensor is attached in the field, the sensor must be placed in an interference-free arrangement with respect to the Type S pitot tube openings (see Method 2, Figure 2-7). As a second alternative, if a difference of not more than 1 percent in the average velocity measurement is to be introduced, the temperature gauge need not be attached to the probe or pitot tube. (This alternative is subject to the approval of the Administrator.)

2.2 Sample Recovery. The following items are needed.

2.2.1 Probe-Liner and Probe-Nozzle Brushes. Nylon bristle brushes with stainless steel wire handles. The probe brush shall have extensions (at least as long as the probe) of stainless steel, Nylon, Teflon, or similarly inert material. The brushes shall be properly sized and shaped to brush out the probe liner and nozzle.

2.2.2 Wash Bottles—Two. Glass wash bottles are recommended; polyethylene wash bottles may be used at the option of the tester. It is recommended that acetone not be stored in polyethylene bottles for longer than a month.

2.2.3 Glass Sample Storage Containers. Chemically resistant, borosilicate glass bottles, for acetone washes, 500 ml or 1000 ml. Screw cap liners shall either be rubber-backed Teflon or shall be constructed so as to be leak-free and resistant to chemical attack by acetone. (Narrow mouth glass bottles have been found to be less prone to leakage.) Alternatively, polyethylene bottles may be used.

2.2.4 Petri Dishes. For filter samples, glass or polyethylene, unless otherwise specified by the Administrator.

2.2.5 Graduated Cylinder and/or Balance. To measure condensed water to within 1 ml or 1 g. Graduated cylinders shall have subdivisions no greater than 2 ml. Most laboratory balances are capable of weighing to the nearest 0.5 g or less. Any of these balances is suitable for use here and in Section 2.3.4.

2.2.6 Plastic Storage Containers. Air-tight containers to store silica gel.

2.2.7 Funnel and Rubber Policeman. To aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

2.2.8 Funnel. Glass or polyethlene, to aid in sample recovery.

2.3 Analysis. For analysis, the following equipment is needed.

2.3.1 Glass Weighing Dishes.

2.3.2 Desiccator.

2.3.3 Analytical Balance. To measure to within 0.1 mg.

2.3.4 Balance. To measure to within 0.5 g.

2.3.5 Beakers. 250 ml.

2.3.6 Hygrometer. To measure the relative humidity of the laboratory environment.

2.3.7 Temperature Gauge. To measure the temperature of the laboratory environment.

## 3. *Reagents*

3.1 Sampling. The reagents used in sampling are as follows:

3.1.1 Filters. Glass fiber filters, without organic binder, exhibiting at least 99.95 percent efficiency ($\leq 0.05$ percent penetration) on 0.3-micron dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM standard method D 2986-71. Test data from the supplier's quality control program are sufficient for this purpose.

3.1.2. Silica Gel. Indicating type, 6 to 16 mesh. If previously used, dry at 175° C (350° F) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to the approval of the Administrator.

3.1.3 Water. When analysis of the material caught in the impingers is required, distilled water shall be used. Run blanks prior to field use to eliminate a high blank on test samples.

3.1.4 Crushed Ice.

3.1.5 Stopcock Grease. Acetone-insoluble, heat-stable silicone grease. This is not necessary if screw-on connectors with Teflon sleeves, or similar, are used. Alternatively, other types of stopcock grease may be used, subject to the approval of the Administrator.

3.2 Sample Recovery. Acetone—reagent grade, $\leq 0.001$ percent residue, in glass bottles—is required. Acetone from metal containers generally has a high residue blank and should not be used. Sometimes, suppliers transfer acetone to glass bottles from metal containers; thus, acetone blanks shall be run prior to field use and only acetone with low blank values ($\leq 0.001$ percent) shall be used. In no case shall a blank value of greater than 0.001 percent of the weight of acetone used be subtracted from the sample weight.

3.3 Analysis. Two reagents are required for the analysis:

3.3.1 Acetone. Same as 3.2.

3.3.2 Desiccant. Anhydrous calcium sulfate, indicating type. Alternatively, other types of desiccants may be used, subject to the approval of the Administrator.

## 4. *Procedure*

4.1 Sampling. The complexity of this method is such that, in order to obtain reliable results, testers should be trained and experienced with the test procedures.

4.1.1 Pretest Preparation. All the components shall be maintained and calibrated according to the procedure described in APTD-0576, unless otherwise specified herein.

Weigh several 200 to 300 g portions of silica gel in air-tight containers to the nearest 0.5 g. Record the total weight of the silica gel plus container, on each container. As an alternative, the silica gel need not be preweighed, but may be weighed directly in its impinger or sampling holder just prior to train assembly.

Check filters visually against light for irregularities and flaws or pinhole leaks. Label filters of the proper diameter on the back side near the edge using numbering machine ink. As an alternative, label the shipping containers (glass or plastic petri dishes) and keep the filters in these containers at all times except during sampling and weighing.

Desiccate the filters at 20±5.6° C (68±10° F) and ambient pressure for at least 24 hours and weigh at intervals of at least 6 hours to a constant weight, i.e., $\leq 0.5$ mg change from previous weighing; record results to the nearest 0.1 mg. During each weighing the filter must not be exposed to the laboratory atmosphere for a period greater than 2 minutes and a relative humidity above 50 percent. Alternatively (unless otherwise specified by the Administrator), the filters may be oven dried at 105° C (220° F) for 2 to 3 hours, desiccated for 2 hours, and weighed. Procedures other than those described, which account for relative humidity effects, may be used, subject to the approval of the Administrator.

4.1.2 Preliminary Determinations. Select the sampling site and the minimum number of sampling points according to Method 1 or as specified by the Administrator. Determine the stack pressure, temperature, and the range of velocity heads using Method 2; it is recommended that a leak-check of the pitot lines (see Method 2, Section 3.1) be performed. Determine the moisture content using Approximation Method 4 or its alternatives for the purpose of making isokinetic sampling rate settings. Determine the stack gas dry molecular weight, as described in Method 2, Section 3.6; if integrated Method 3 sampling is used for molecular weight determination, the integrated bag sample shall be taken simultaneously with, and for the same total length of time as, the particulate sample run.

Select a nozzle size based on the range of velocity heads, such that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle size. Ensure that the proper differential pressure gauge is chosen for the range of velocity heads encountered (see Section 2.2 of Method 2).

Select a suitable probe liner and probe length such that all traverse points can be sampled. For large stacks, consider sampling from opposite sides of the stack to reduce the length of probes.

Select a total sampling time greater than or equal to the minimum total sampling time specified in the test procedures for the specific industry such that (1) the sampling time per point is not less than 2 min (or some greater time interval as specified by the Administrator), and (2) the sample volume taken (corrected to standard conditions) will exceed the required minimum total gas sample volume. The latter is based on an approximate average sampling rate.

It is recommended that the number of minutes sampled at each point be an integer or an integer plus one-half minute, in order to avoid timekeeping errors.

In some circumstances, e.g., batch cycles, it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes. In these cases, the Administrator's approval must first be obtained.

4.1.3 Preparation of Collection Train. During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to begin.

Place 100 ml of water in each of the first two impingers, leave the third impinger empty, and transfer approximately 200 to 300 g of preweighed silica gel from its container to the fourth impinger. More silica gel may be used, but care should be taken to ensure that it is not entrained and carried out from the impinger during sampling. Place the container in a clean place for later use in the sample recovery. Alternatively, the weight of the silica gel plus impinger may be determined to the nearest 0.5 g and recorded.

Using a tweezer or clean disposable surgical gloves, place a labeled (identified) and weighed filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as to prevent the sample gas stream from circumventing the filter. Check the filter for tears after assembly is completed.

When glass liners are used, install the selected nozzle using a Viton A O-ring when stack temperatures are less than 260° C (500° F) and an asbestos string gasket when temperatures are higher. See APTD-0576 for details. Other connecting systems using either 316 stainless steel or Teflon ferrules may be used. When metal liners are used, install the nozzle as above or by a leak-free direct mechanical connection. Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

Set up the train as in Figure 5-1, using (if necessary) a very light coat of silicone grease on all ground glass joints, greasing only the outer portion (see APTD-0576) to avoid possibility of contamination by the silicone grease. Subject to the approval of the Administrator, a glass cyclone may be used between the probe and filter holder when the total particulate catch is expected to exceed 100 mg or when water droplets are present in the stack gas.

Place crushed ice around the impingers.

4.1.4 Leak-Check Procedures.

4.1.4.1 Pretest Leak-Check. A pretest leak-check is recommended, but not required. If the tester opts to conduct the pretest leak-check, the following procedure shall be used.

After the sampling train has been assembled, turn on and set the filter and probe heating systems at the desired operating temperatures. Allow time for the temperatures to stabilize. If a Viton A O-ring or other leak-free connection is used in assembling the probe nozzle to the probe liner, leak-check the train at the sampling site by plugging the nozzle and pulling a 380 mm Hg (15 in. Hg) vacuum.

NOTE.—A lower vacuum may be used, provided that it is not exceeded during the test.

If an asbestos string is used, do not connect the probe to the train during the leak-check. Instead, leak-check the train by first plugging the inlet to the filter holder (cyclone, if applicable) and pulling a 380 mm Hg (15 in. Hg) vacuum (see Note immediately above). Then connect the probe to the train and leak-check at about 25 mm Hg (1 in. Hg) vacuum; alternatively, the probe may be leak-checked with the rest of the sampling train, in one step, at 380 mm Hg (15 in. Hg) vacuum. Leakage rates in excess of 4 percent of the average sampling rate or 0.00057 $m^3$/min (0.02 cfm), whichever is less, are unacceptable.

The following leak-check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with bypass valve fully open and coarse adjust valve completely closed. Partially open the coarse adjust valve and slowly close the bypass valve until the desired vacuum is reached. Do not reverse direction of bypass valve; this will cause water to back up into the filter holder. If the desired vacuum is exceeded, either leak-check at this higher vacuum or end the leak check as shown below and start over.

When the leak-check is completed, first slowly remove the plug from the inlet to the probe, filter holder, or cyclone (if applicable) and immediately turn off the vacuum pump. This prevents the water in the impingers from being forced backward into the filter holder and silica gel from being entrained backward into the third impinger.

4.1.4.2 Leak-Checks During Sample Run. If, during the sampling run, a component (e.g., filter assembly or impinger) change becomes necessary, a leak-check shall be conducted immediately before the change is made. The leak-check shall be done according to the procedure outlined in Section 4.1.4.1 above, except that it shall be done at a vacuum equal to or greater than the maximum value recorded up to that point in the test. If the leakage rate is found to be no greater than 0.00057 $m^3$/min (0.02 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction will need to be applied to the total volume of dry gas metered; if, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and plan to correct the sample volume as shown in Section 6.3 of this method, or shall void the sampling run.

Immediately after component changes, leak-checks are optional; if such leak-checks are done, the procedure outlined in Section 4.1.4.1 above shall be used.

4.1.4.3 Post-test Leak-Check. A leak-check is mandatory at the conclusion of each sampling run. The leak-check shall be done in accordance with the procedures outlined in Section 4.1.4.1, except that it shall be conducted at a vacuum equal to or greater than the maximum value reached during the sampling run. If the leakage rate is found to be no greater than 0.00057 $m^3$/min (0.02 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction need be applied to the total volume of dry gas metered. If, however, a higher leakago rate is obtained, the tester shall either record the leakage rate and correct the sample volume as shown in Section 6.3 of this method, or shall void the sampling run.

4.1.5 Particulate Train Operation. During the sampling run, maintain an isokinetic sampling rate (within 10 percent of true isokinetic unless otherwise specified by the Administrator) and a temperature around the filter of 120±14° C (248±25° F), or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator.

For each run, record the data required on a data sheet such as the one shown in Figure 5-2. Be sure to record the initial dry gas meter reading. Record the dry gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made, before and after each leak check, and when sampling is halted. Take other readings required by Figure 5-2 at least once at each sample point during each time increment and additional readings when significant changes (20 percent variation in velocity head readings) necessitate additional adjustments in flow rate. Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse.

Clean the portholes prior to the test run to minimize the chance of sampling deposited material. To begin sampling, remove the nozzle cap, verify that the filter and probe heating systems are up to temperature, and that the pitot tube and probe are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs are available, which aid in the rapid adjustment of the isokinetic sampling rate without excessive computations. These nomographs are designed for use when the Type S pitot tube coefficient is 0.85±0.02, and the stack gas equivalent density (dry molecular weight) is equal to 29±4. APTD-0576 details the procedure for using the nomographs. If $C_p$ and $M_d$ are outside the above stated ranges do not use the nomographs unless appropriate steps (see Citation 7 in Section 7) are taken to compensate for the deviations.

When the stack is under significant negative pressure (height of impinger stem), take care to close the coarse adjust valve before inserting the probe into the stack to prevent water from backing into the filter holder. If necessary, the pump may be turned on with the coarse adjust valve closed.

When the probe is in position, block off the openings around the probe and porthole to prevent unrepresentative dilution of the gas stream.

Traverse the stack cross-section, as required by Method 1 or as specified by the Administrator, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the portholes; this minimizes the chance of extracting deposited material.

During the test run, make periodic adjustments to keep the temperature around the filter holder at the proper level; add more ice and, if necessary, salt to maintain a temperature of less than 20° C (68° F) at the condenser/silica gel outlet. Also, periodically check the level and zero of the manometer.

If the pressure drop across the filter becomes too high, making isokinetic sampling difficult to maintain, the filter may be replaced in the midst of a sample run. It is recommended that another complete filter assembly be used rather than attempting to change the filter itself. Before a new filter assembly is installed, conduct a leak-check (see Section 4.1.4.2). The total particulate weight shall include the summation of all filter assembly catches.

A single train shall be used for the entire sample run, except in cases where simultaneous sampling is required in two or more separate ducts or at two or more different locations within the same duct, or, in cases where equipment failure necessitates a change of trains. In all other situations, the use of two or more trains will be subject to the approval of the Administrator.

Note that when two or more trains are used, separate analyses of the front-half and (if applicable) impinger catches from each train shall be performed, unless identical nozzle sizes were used on all trains, in which case, the front-half catches from the individual trains may be combined (as may the impinger catches) and one analysis of front-half catch and one analysis of impinger catch may be performed. Consult with the Administrator for details concerning the calculation of results when two or more trains are used.

At the end of the sample run, turn off the coarse adjust valve, remove the probe and nozzle from the stack, turn off the pump, record the final dry gas meter reading, and conduct a post-test leak-check, as outlined in Section 4.1.4.3. Also, leak-check the pitot lines as described in Method 2, Section 3.1; the lines must pass this leak-check, in order to validate the velocity head data.

4.1.6 Calculation of Percent Isokinetic. Calculate percent isokinetic (see Calculations, Section 6) to determine whether the run was valid or another test run should be made. If there was difficulty in maintaining isokinetic rates due to source conditions, consult with the Administrator for possible variance on the isokinetic rates.

4.2 Sample Recovery. Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period. Allow the probe to cool.

When the probe can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it to prevent losing or gaining particulate matter. Do not cap off the probe tip tightly while the sampling train is cooling down as this would create a vacuum in the filter holder, thus drawing water from the impingers into the filter holder.

Before moving the sample train to the cleanup site, remove the probe from the sample train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate that might be present. Wipe off the silicone grease from the filter inlet where the probe was fastened and cap it. Remove the umbilical cord from the last impinger and cap the impinger. If a flexible line is used between the first impinger or condenser and the filter holder, disconnect the line at the

PLANT ____________
LOCATION ____________
OPERATOR ____________
DATE ____________
RUN NO. ____________
SAMPLE BOX NO. ____________
METER BOX NO. ____________
METER $\Delta H_@$ ____________
C FACTOR ____________
PITOT TUBE COEFFICIENT, $C_p$ ____________

SCHEMATIC OF STACK CROSS SECTION

AMBIENT TEMPERATURE ____________
BAROMETRIC PRESSURE ____________
ASSUMED MOISTURE, % ____________
PROBE LENGTH, m (ft) ____________
NOZZLE IDENTIFICATION NO. ____________
AVERAGE CALIBRATED NOZZLE DIAMETER, cm (in.) ____________
PROBE HEATER SETTING ____________
LEAK RATE, $m^3$/min.(cfm) ____________
PROBE LINER MATERIAL ____________
STATIC PRESSURE, mm Hg (in. Hg) ____________
FILTER NO. ____________

| TRAVERSE POINT NUMBER | SAMPLING TIME ($\theta$), min. | VACUUM mm Hg (in. Hg) | STACK TEMPERATURE ($T_S$) °C (°F) | VELOCITY HEAD ($\Delta P_S$), mm(in.)$H_2O$ | PRESSURE DIFFERENTIAL ACROSS ORIFICE METER mm $H_2O$ (in. $H_2O$) | GAS SAMPLE VOLUME $m^3$ ($ft^3$) | GAS SAMPLE TEMPERATURE AT DRY GAS METER | | FILTER HOLDER TEMPERATURE, °C (°F) | TEMPERATURE OF GAS LEAVING CONDENSER OR LAST IMPINGER, °C (°F) |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | INLET °C (°F) | OUTLET °C (°F) | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| TOTAL | | | | | | | Avg. | Avg. | | |
| AVERAGE | | | | | | | Avg. | | | |

Figure 5-2. Particulate field data.

**Plant**________________________________________

**Date**________________________________________

**Run No.** ______________________________________

**Filter No.** ______________________________________

**Amount liquid lost during transport** ____________________

**Acetone blank volume, ml**____________________________

**Acetone wash volume, ml**_____________________________

**Acetone blank concentration, mg/mg (equation 5-4)**________________

**Acetone wash blank, mg (equation 5-5)** ____________________

| CONTAINER NUMBER | WEIGHT OF PARTICULATE COLLECTED, mg | | |
|---|---|---|---|
| | FINAL WEIGHT | TARE WEIGHT | WEIGHT GAIN |
| 1 | | | |
| 2 | | | |
| TOTAL | ☒ | ☒ | |
| | | Less acetone blank | |
| | | Weight of particulate matter | |

| | VOLUME OF LIQUID WATER COLLECTED | | |
|---|---|---|---|
| | IMPINGER VOLUME, ml. | SILICA GEL WEIGHT, g | |
| FINAL | | | |
| INITIAL | | | |
| LIQUID COLLECTED | | | |
| TOTAL VOLUME COLLECTED | | g* | ml |

* CONVERT WEIGHT OF WATER TO VOLUME BY DIVIDING TOTAL WEIGHT INCREASE BY DENSITY OF WATER (1g/ml)

$$\frac{\text{INCREASE, g}}{\text{1 g/ml}} = \text{VOLUME WATER, ml}$$

Figure 5-3. Analytical data.

filter holder and let any condensed water or liquid drain into the impingers or condenser. After wiping off the silicone grease, cap off the filter holder outlet and impinger inlet. Either ground-glass stoppers, plastic caps, or serum caps may be used to close these openings.

Transfer the probe and filter-impinger assembly to the cleanup area. This area should be clean and protected from the wind so that the chances of contaminating or losing the sample will be minimized.

Save a portion of the acetone used for cleanup as a blank. Take 200 ml of this acetone directly from the wash bottle being used and place it in a glass sample container labeled "acetone blank."

Inspect the train prior to and during disassembly and note any abnormal conditions. Treat the samples as follows:

*Container No. 1.* Carefully remove the filter from the filter holder and place it in its identified petri dish container. Use a pair of tweezers and/or clean disposable surgical gloves to handle the filter. If it is necessary to fold the filter, do so such that the particulate cake is inside the fold. Carefully transfer to the petri dish any particulate matter and/or filter fibers which adhere to the filter holder gasket, by using a dry nylon bristle brush and/or a sharp-edged blade. Seal the container.

*Container No. 2.* Taking care to see that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover particulate matter or any condensate from the probe nozzle, probe fitting, probe liner, and front half of the filter holder by washing these components with acetone and placing the wash in a glass container. Distilled water may be used instead of acetone when approved by the Administrator and shall be used when specified by the Administrator, in these cases, save a water blank and follow the Administrator's directions on analysis. Perform the acetone rinses as follows:

Carefully remove the probe nozzle and clean the inside surface by rinsing with acetone from a wash bottle and brushing with a nylon bristle brush. Brush until the acetone rinse shows no visible particles, after which make a final rinse of the inside surface with acetone.

Brush and rinse the inside parts of the Swagelok fitting with acetone in a similar way until no visible particles remain.

Rinse the probe liner with acetone by tilting and rotating the probe while squirting acetone into its upper end so that all inside surfaces will be wetted with acetone. Let the acetone drain from the lower end into the sample container. A funnel (glass or polyethylene) may be used to aid in transferring liquid washes to the container. Follow the acetone rinse with a probe brush. Hold the probe in an inclined position, squirt acetone into the upper end as the probe brush is being pushed with a twisting action through the probe; hold a sample container underneath the lower end of the probe, and catch any acetone and particulate matter which is brushed from the probe. Run the brush through the probe three times or more until no visible particulate matter is carried out with the acetone or until none remains in the probe liner on visual inspection. With stainless steel or other metal probes, run the brush through in the above prescribed manner at least six times since metal probes have small crevices in which particulate matter can be entrapped. Rinse the brush with acetone, and quantitatively collect these washings in the sample container. After the brushing, make a final acetone rinse of the probe as described above.

It is recommended that two people be used to clean the probe to minimize sample losses. Between sampling runs, keep brushes clean and protected from contamination.

After ensuring that all joints have been wiped clean of silicone grease, clean the inside of the front half of the filter holder by rubbing the surfaces with a nylon bristle brush and rinsing with acetone. Rinse each surface three times or more if needed to remove visible particulate. Make a final rinse of the brush and filter holder. Carefully rinse out the glass cyclone, also (if applicable). After all acetone washings and particulate matter have been collected in the sample container, tighten the lid on the sample container so that acetone will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether or not leakage occurred during transport. Label the container to clearly identify its contents.

*Container No. 3.* Note the color of the indicating silica gel to determine if it has been completely spent and make a notation of its condition. Transfer the silica gel from the fourth impinger to its original container and seal. A funnel may make it easier to pour the silica gel without spilling. A rubber policeman may be used as an aid in removing the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that may adhere to the impinger wall and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, follow the procedure for container No. 3 in Section 4.3.

*Impinger Water.* Treat the impingers as follows; Make a notation of any color or film in the liquid catch. Measure the liquid which is in the first three impingers to within ±1 ml by using a graduated cylinder or by weighing it to within ±0.5 g by using a balance (if one is available). Record the volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas.

Discard the liquid after measuring and recording the volume or weight, unless analysis of the impinger catch is required (see Note, Section 2.1.7).

If a different type of condenser is used, measure the amount of moisture condensed either volumetrically or gravimetrically.

Whenever possible, containers should be shipped in such a way that they remain upright at all times.

4.3 Analysis. Record the data required on a sheet such as the one shown in Figure 5-3. Handle each sample container as follows:

*Container No. 1.* Leave the contents in the shipping container or transfer the filter and any loose particulate from the sample container to a tared glass weighing dish. Desiccate for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh to a constant weight and report the results to the nearest 0.1 mg. For purposes of this Section, 4.3, the term "constant weight" means a difference of no more than 0.5 mg or 1 percent of total weight less tare weight, whichever is greater, between two consecutive weighings, with no less than 6 hours of desiccation time between weighings.

Alternatively, the sample may be oven dried at 105° C (220° F) for 2 to 3 hours, cooled in the desiccator, and weighed to a constant weight, unless otherwise specified by the Administrator. The tester may also opt to oven dry the sample at 105° C (220° F) for 2 to 3 hours, weigh the sample, and use this weight as a final weight.

*Container No. 2.* Note the level of liquid in the container and confirm on the analysis sheet whether or not leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in this container either volumetrically to ±1 ml or gravimetrically to ±0.5 g. Transfer the contents to a tared 250-ml beaker and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours and weigh to a constant weight. Report the results to the nearest 0.1 mg.

*Container No. 3.* Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance. This step may be conducted in the field.

*"Acetone Blank" Container.* Measure acetone in this container either volumetrically or gravimetrically. Transfer the acetone to a tared 250-ml beaker and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours and weigh to a contsant weight. Report the results to the nearest 0.1 mg.

NOTE.—At the option of the tester, the contents of Container No. 2 as well as the acetone blank container may be evaporated at temperatures higher than ambient. If evaporation is done at an elevated temperature, the temperature must be below the boiling point of the solvent; also, to prevent "bumping," the evaporation process must be closely supervised, and the contents of the beaker must be swirled occasionally to maintain an even temperature. Use extreme care, as acetone is highly flammable and has a low flash point.

### 5. *Calibration*

Maintain a laboratory log of all calibrations.

5.1 Probe Nozzle. Probe nozzles shall be calibrated before their initial use in the field. Using a micrometer, measure the inside diameter of the nozzle to the nearest 0.025 mm (0.001 in.). Make three separate measurements using different diameters each time, and obtain the average of the measurements. The difference between the high and low numbers shall not exceed 0.1 mm (0.004 in.). When nozzles become nicked, dented, or corroded, they shall be reshaped, sharpened, and recalibrated before use. Each nozzle shall be permanently and uniquely identified.

5.2 Pitot Tube. The Type S pitot tube assembly shall be calibrated according to the procedure outlined in Section 4 of Method 2.

5.3 Metering System. Before its initial use in the field, the metering system shall be calibrated according to the procedure outlined in APTD-0576. Instead of physically adjusting the dry gas meter dial readings to correspond to the wet test meter readings, calibration factors may be used to mathematically correct the gas meter dial readings to the proper values. Before calibrating the metering system, it is suggested that a leak-check be conducted. For metering systems having diaphragm pumps, the normal leak-check procedure will not detect leakages within the pump. For these cases the following leak-check procedure is suggested: make a 10-minute calibration run at 0.00057 m $^3$/min (0.02 cfm); at the end of the run, take the difference of the measured wet test meter and dry gas meter volumes; divide the difference by 10, to get the leak rate. The leak rate should not exceed 0.00057 m $^3$/min (0.02 cfm).

After each field use, the calibration of the metering system shall be checked by performing three calibration runs at a single, intermediate orifice setting (based on the previous field test), with the vacuum set at the maximum value reached during the test series. To adjust the vacuum, insert a valve between the wet test meter and the inlet of the metering system. Calculate the average value of the calibration factor. If the calibration has changed by more than 5 percent, recalibrate the meter over the full range of orifice settings, as outlined in APTD-0576.

Alternative procedures, e.g., using the orifice meter coefficients, may be used, subject to the approval of the Administrator.

NOTE.—If the dry gas meter coefficient values obtained before and after a test series differ by more than 5 percent, the test series shall either be voided, or calculations for the test series shall be performed using whichever meter coefficient value (i.e., before or after) gives the lower value of total sample volume.

Reproduced from
best available copy.

5.4 Probe Heater Calibration. The probe heating system shall be calibrated before its initial use in the field according to the procedure outlined in APTD-0576. Probes constructed according to APTD-0581 need not be calibrated if the calibration curves in APTD-0576 are used.

5.5 Temperature Gauges. Use the procedure in Section 4.3 of Method 2 to calibrate in-stack temperature gauges. Dial thermometers, such as are used for the dry gas meter and condenser outlet, shall be calibrated against mercury-in-glass thermometers.

5.6 Leak Check of Metering System Shown in Figure 5-1. That portion of the sampling train from the pump to the orifice meter should be leak checked prior to initial use and after each shipment. Leakage after the pump will result in less volume being recorded than is actually sampled. The following procedure is suggested (see Figure 5-4): Close the main valve on the meter box. Insert a one-hole rubber stopper with rubber tubing attached into the orifice exhaust pipe. Disconnect and vent the low side of the orifice manometer. Close off the low side orifice tap. Pressurize the system to 13 to 18 cm (5 to 7 in.) water column by blowing into the rubber tubing. Pinch off the tubing and observe the manometer for one minute. A loss of pressure on the manometer indicates a leak in the meter box; leaks, if present, must be corrected.

5.7 Barometer. Calibrate against a mercury barometer.

### 6. *Calculations*

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after the final calculation. Other forms of the equations may be used as long as they give equivalent results.

6.1 Nomenclature

$A_n$ = Cross-sectional area of nozzle, m² (ft²).
$B_{ws}$ = Water vapor in the gas stream, proportion by volume.
$C_a$ = Acetone blank residue concentrations, mg/g.
$c_s$ = Concentration of particulate matter in stack gas, dry basis, corrected to standard conditions, g/dscm (g/dscf).
$I$ = Percent of isokinetic sampling.
$L_a$ = Maximum acceptable leakage rate for either a pretest leak check or for a leak check following a component change; equal to 0.00057 m³/min (0.02 cfm) or 4 percent of the average sampling rate, whichever is less.
$L_i$ = Individual leakage rate observed during the leak check conducted prior to the "$i^{th}$" component change ($i$=1, 2, 3 . . . . $n$), m³/min (cfm).
$L_p$ = Leakage rate observed during the post-test leak check, m³/min (cfm).
$m_n$ = Total amount of particulate matter collected, mg.
$M_w$ = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole).
$m_a$ = Mass of residue of acetone after evaporation, mg.
$P_{bar}$ = Barometric pressure at the sampling site, mm Hg (in. Hg).
$P_s$ = Absolute stack gas pressure, mm Hg (in. Hg).
$P_{std}$ = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).
$R$ = Ideal gas constant, 0.06236 mm Hg-m³/°K-g-mole (21.85 in. Hg-ft³/°R-lb-mole).
$T_m$ = Absolute average dry gas meter temperature (see Figure 5-2), °K (°R).
$T_s$ = Absolute average stack gas temperature (see Figure 5-2), °K (°R).
$T_{std}$ = Standard absolute temperature, 293° K (528° R).
$V_a$ = Volume of acetone blank, ml.
$V_{aw}$ = Volume of acetone used in wash, ml.
$V_{lc}$ = Total volume of liquid collected in impingers and silica gel (see Figure 5-3), ml.
$V_m$ = Volume of gas sample as measured by dry gas meter, dcm (dcf).
$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter, corrected to standard conditions, dscm (dscf).
$V_{w(std)}$ = Volume of water vapor in the gas sample, corrected to standard conditions, scm (scf).
$V_s$ = Stack gas velocity, calculated by Method 2, Equation 2-9, using data obtained from Method 5, m/sec (ft/sec).
$W_a$ = Weight of residue in acetone wash, mg.
$Y$ = Dry gas meter calibration factor.
$\Delta H$ = Average pressure differential across the orifice meter (see Figure 5-2), mm $H_2O$ (in. $H_2O$).
$\rho_a$ = Density of acetone, mg/ml (see label on bottle).
$\rho_w$ = Density of water, 0.9982 g/ml (0.002201 lb/ml).
$\theta$ = Total sampling time, min.
$\theta_1$ = Sampling time interval, from the beginning of a run until the first component change, min.
$\theta_i$ = Sampling time interval, between two successive component changes, beginning with the interval between the first and second changes, min.
$\theta_p$ = Sampling time interval, from the final ($n^{th}$) component change until the end of the sampling run, min.
13.6 = Specific gravity of mercury.
60 = Sec/min.
100 = Conversion to percent.

6.2 Average dry gas meter temperature and average orifice pressure drop. See data sheet (Figure 5-2).

6.3 Dry Gas Volume. Correct the sample volume measured by the dry gas meter to standard conditions (20° C, 760 mm Hg or 68° F, 29.92 in. Hg) by using Equation 5-1.

$$V_{m(std)} = V_m Y \left(\frac{T_{std}}{T_m}\right) \left[\frac{P_{bar} + \frac{\Delta H}{13.6}}{P_{std}}\right]$$

$$= K_1 V_m Y \frac{P_{bar} + (\Delta H/13.6)}{T_m}$$

Equation 5-1

where:

$m_1$ = 0.3858 °K/mm Hg for metric units
= 17.64 °R/in. Hg for English units

NOTE.—Equation 5-1 can be used as written unless the leakage rate observed during any of the mandatory leak checks (i.e., the post-test leak check or leak checks conducted prior to component changes) exceeds $L_a$. If $L_p$ or $L_i$ exceeds $L_a$, Equation 5-1 must be modified as follows;

(a) Case I. No component changes made during sampling run. In this case, replace $V_m$ in Equation 5-1 with the expression;

$$V_m - (L_p - L_a)\theta]$$

(b) Case II. One or more component changes made during the sampling run. In this case, replace $V_m$ in Equation 5-1 by the expression:

$$\left[V_m - (L_1 - L_a)\theta_1 - \sum_{i=2}^{n} (L_i - L_a)\theta_i - (L_p - L_a)\theta_p\right]$$

and substitute only for those leakage rates ($L_i$ or $L_p$) which exceed $L_a$.

6.4 Volume of water vapor.

Equation 5-2

$$V_{w(std)} = V_{lc} \left(\frac{\rho_w}{M_w}\right)\left(\frac{R T_{std}}{P_{std}}\right) = K_2 V_{lc}$$

where:

$K_2$ = 0.001333 m³/ml for metric units
= 0.04707 ft³/ml for English units.

6.5 Moisture Content.

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}}$$

Equation 5-3

NOTE.—In saturated or water droplet-laden gas streams, two calculations of the moisture content of the stack gas shall be made, one from the impinger analysis (Equation 5-3), and a second from the assumption of saturated conditions. The lower of the two values of $B_{ws}$ shall be considered correct. The procedure for determining the moisture content based upon assumption of saturated conditions is given in the Note of Section 1.2 of Method 4. For the purposes of this method, the average stack gas temperature from Figure 5-2 may be used to make this determination, provided that the accuracy of the in-stack temperature sensor is ± 1° C (2° F).

6.6 Acetone Blank Concentration.

$$C_a = \frac{m_a}{V_a \rho_a}$$

Equation 5-4

6.7 Acetone Wash Blank.

$$W_a = C_a V_{aw} \rho_a$$

Equation 5-5

6.8 Total Particulate Weight. Determine the total particulate catch from the sum of the weights obtained from containers 1 and 2 less the acetone blank (see Figure 5-3). NOTE.—Refer to Section 4.1.5 to assist in calculation of results involving two or more filter assemblies or two or more sampling trains.

6.9 Particulate Concentration.

$$c_s = (0.001\ g/mg)\ (m_n/V_{m(std)})$$

Equation 5-6

6.10 Conversion Factors:

| From | To | Multiply by |
|---|---|---|
| scf | m³ | 0.02832 |
| g/ft³ | gr/ft³ | 15.43 |
| g/ft³ | lb/ft³ | $2.205 \times 10^{-3}$ |
| g/ft³ | g/m³ | 35.31 |

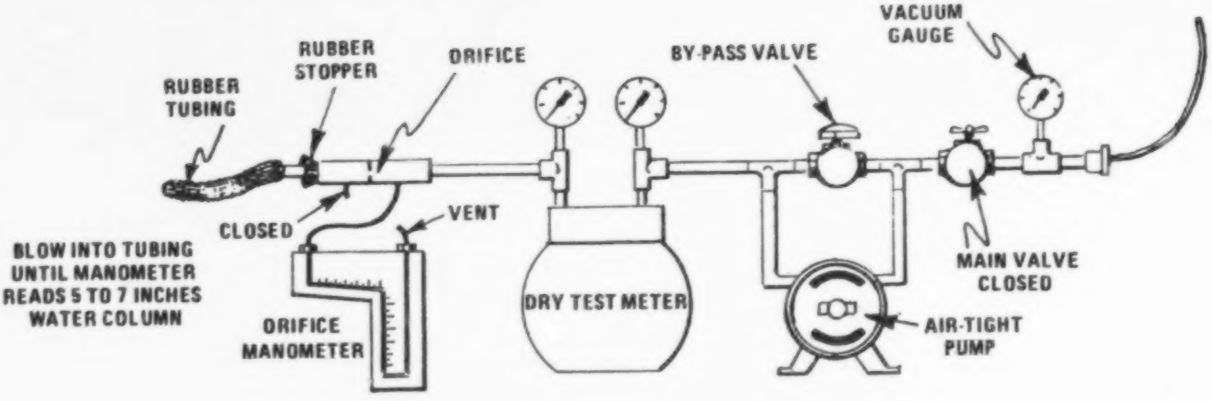

VACUUM GAUGE
RUBBER STOPPER
ORIFICE
BY-PASS VALVE
RUBBER TUBING
CLOSED
VENT
BLOW INTO TUBING UNTIL MANOMETER READS 5 TO 7 INCHES WATER COLUMN
ORIFICE MANOMETER
DRY TEST METER
MAIN VALVE CLOSED
AIR-TIGHT PUMP

Figure 5-4. Leak check of meter box.

**6.11 Isokinetic Variation.**

**6.11.1 Calculation From Raw Data.**

$$I=\frac{100\,T_s[K_3V_{1c}+(V_m/T_m)\,(P_{bar}+\Delta H/13.6)]}{60\,\theta v_s\,P_s\,A_n}$$

Equation 5-7

**where:**

$K_3$=0.003454 mm Hg−m³/ml−°K for metric units.
=0.002669 in. Hg−ft³/ml−°R for English units.

**6.11.2 Calculation From Intermediate Values.**

$$I=\frac{T_sV_{m\,(std)}P_{std}\,100}{T_{std}v_s\theta\,A_nP_s\,60(1-B_{ws})}$$

$$=K_4\,\frac{T_sV_{m\,(std)}}{P_sV_sA_n\theta\,(1-B_{ws})}$$

Equation 5-8

**where:**

$K_4$=4.320 for metric units
=0.09450 for English units.

**6.12 Acceptable Results.** If 90 percent $\leq I \leq$ 110 percent, the results are acceptable. If the results are low in comparison to the standard and *I* is beyond the acceptable range, or, if *I* is less than 90 percent, the Administrator may opt to accept the results. Use Citation 4 to make judgments. Otherwise, reject the results and repeat the test.

7. *Bibliography*

1. Addendum to Specifications for Incinerator Testing at Federal Facilities. PHS, NCAPC. Dec. 6, 1967.

2. Martin, Robert M. Construction Details of Isokinetic Source-Sampling Equipment. Environmental Protection Agency. Research Triangle Park, N.C. APTD-0581. April, 1971.

3. Rom, Jerome J. Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment. Environmental Protection Agency. Research Triangle Park, N.C. APTD-0576. March, 1972.

4. Smith, W. S., R. T. Shigehara, and W. F. Todd. A Method of Interpreting Stack Sampling Data. Paper Presented at the 63d Annual Meeting of the Air Pollution Control Association, St. Louis, Mo. June 14-19, 1970.

5. Smith, W. S., et al. Stack Gas Sampling Improved and Simplified With New Equipment. APCA Paper No. 67-119. 1967.

6. Specifications for Incinerator Testing at Federal Facilities. PHS, NCAPC. 1967.

7. Shigehara, R. T. Adjustments in the EPA Nomograph for Different Pitot Tube Coefficients and Dry Molecular Weights. Stack Sampling News 2:4-11. October, 1974.

8. Vollaro, R. F. A Survey of Commercially Available Instrumentation For the Measurement of Low-Range Gas Velocities. U.S. Environmental Protection Agency, Emission Measurement Branch. Research Triangle Park, N.C. November, 1976 (unpublished paper).

9. Annual Book of ASTM Standards. Part 26. Gaseous Fuels; Coal and Coke; Atmospheric Analysis. American Society for Testing and Materials. Philadelphia, Pa. 1974. pp. 617-622.

# METHOD 6

# DETERMINATION OF SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES

### 1. *Principle and Applicability*

1.1 Principle. A gas sample is extracted from the sampling point in the stack. The sulfuric acid mist (including sulfur trioxide) and the sulfur dioxide are separated. The sulfur dioxide fraction is measured by the barium-thorin titration method.

1.2 Applicability. This method is applicable for the determination of sulfur dioxide emissions from stationary sources. The minimum detectable limit of the method has been determined to be 3.4 milligrams (mg) of $SO_2/m^3$ ($2.12\times10^{-7}$ lb/ft³). Although no upper limit has been established, tests have shown that concentrations as high as 80,000 mg/m³ of $SO_2$ can be collected efficiently in two midget impingers, each containing 15 milliliters of 3 percent hydrogen peroxide, at a rate of 1.0 lpm for 20 minutes. Based on theoretical calculations, the upper concentration limit in a 20-liter sample is about 93,300 mg/m³.

Possible interferents are free ammonia, water-soluble cations, and fluorides. The cations and fluorides are removed by glass wool filters and an isopropanol bubbler, and hence do not affect the $SO_2$ analysis. When samples are being taken from a gas stream with high concentrations of very fine metallic fumes (such as in inlets to control devices), a high-efficiency glass fiber filter must be used in place of the glass wool plug (i.e., the one in the probe) to remove the cation interferents.

Free ammonia interferes by reacting with $SO_2$ to form particulate sulfite and by reacting with the indicator. If free ammonia is present (this can be determined by knowledge of the process and noticing white particulate matter in the probe and isopropanol bubbler), alternative methods, subject to the approval of the Administrator, U.S. Environmental Protection Agency, are required.

### 2. *Apparatus*

2.1 **Sampling.** The sampling train is shown in Figure 6-1, and component parts are discussed below. The tester has the option of substituting sampling equipment described in Method 8 in place of the midget impinger equipment of Method 6. However, the Method 8 train must be modified to include a heated filter between the probe and isopropanol impinger, and the operation of the sampling train and sample analysis must be at the flow rates and solution volumes defined in Method 8.

The tester also has the option of determining $SO_2$ simultaneously with particulate matter and moisture determinations by (1) replacing the water in a Method 5 impinger system with 3 percent perioxide solution, or (2) by replacing the Method 5 water impinger system with a Method 8 isopropanol-filter-peroxide system. The analysis for $SO_2$ must be consistent with the procedure in Method 8.

2.1.1 Probe. Borosilicate glass, or stainless steel (other materials of construction may be used, subject to the approval of the Administrator), approximately 6-mm inside diameter, with a heating system to prevent water condensation and a filter (either in-stack or heated out-stack) to remove particulate matter, including sulfuric acid mist. A plug of glass wool is a satisfactory filter.

2.1.2 Bubbler and Impingers. One midget bubbler, with medium-coarse glass frit and borosilicate or quartz glass wool packed in top (see Figure 6-1) to prevent sulfuric acid mist carryover, and three 30-ml midget impingers. The bubbler and midget impingers must be connected in series with leak-free glass connectors. Silicone grease may be used, if necessary, to prevent leakage.

At the option of the tester, a midget impinger may be used in place of the midget bubbler.

Other collection absorbers and flow rates may be used, but are subject to the approval of the Administrator. Also, collection efficiency must be shown to be at least 99 percent for each test run and must be documented in the report. If the efficiency is found to be acceptable after a series of three tests, further documentation is not required. To conduct the efficiency test, an extra absorber must be added and analyzed separately. This extra absorber must not contain more than 1 percent of the total $SO_2$.

2.1.3 Glass Wool. Borosilicate or quarts.

2.1.4 Stopcock Grease. Acetone-insoluble, heat-stable silicone grease may be used, if necessary.

2.1.5 Temperature Gauge. Dial thermometer, or equivalent, to measure temperature of gas leaving impinger train to within 1° C (2° F.)

2.1.6 Drying Tube. Tube packed with 6- to 16-mesh indicating type silica gel, or equivalent, to dry the gas sample and to protect the meter and pump. If the sillac gel has been used previously, dry at 175° C (350° F) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to approval of the Administrator.

2.1.7 Value. Needle value, to regulate sample gas flow rate.

2.1.8 Pump. Leak-free diaphragm pump, or equivalent, to pull gas through the train. Install a small tank between the pump and rate meter to eliminate the pulsation effect of the diaphragm pump on the rotameter.

2.1.9 Rate Meter. Rotameter, or equivalent, capable of measuring flow rate to within 2 percent of the selected flow rate of about 1000 cc/min.

2.1.10 Volume Meter. Dry gas meter, sufficiently accurate to measure the sample volume within 2 percent, calibrated at the selected flow rate and conditions actually encountered during sampling, and equipped with a temperature gauge (dial thermometer, or equivalent) capable of measuring temperature to within 3°C (5.4°F ).

2.1.11 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby national weather service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

2.1.12 Vacuum Gauge. At least 760 mm Hg (30 in. Hg) gauge, to be used for leak check of the sampling train.

2.2 Sample Recovery.

2.2.1 Wash bottles. Polyethylene or glass, 500 ml, two.

2.2.2 Storage Bottles. Polyethylene, 100 ml, to store impinger samples (one per sample).

2.3 Analysis.

2.3.1 Pipettes. Volumetric type, 5-ml, 20-ml (one per sample), and 25-ml sizes.

2.3.2 Volumetric Flasks. 100-ml size (one per sample) and 100-ml size.

2.3.3 Burettes. 5- and 50-ml sizes.

2.3.4 Erlenmeyer Flasks. 250 ml-size (one for each sample, blank, and standard).

2.3.5 Dropping Bottle. 125-ml size, to add indicator.

2.3.6 Graduated Cylinder. 100-ml size.

2.3.7 Spectrophotometer. To measure absorbance at 352 nanometers.

### 3. *Reagents*

Unless otherwise indicated, all reagents must conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society. Where such specifications are not available, use the best available grade.

3.1 Sampling.

3.1.1 Water. Deionized, distilled to conform to ASTM specification D1193-74, Type 3. At the option of the analyst, the $KMnO_4$ test for oxidizable organic matter may be omitted when high concentrations of organic matter are not expected to be present.

3.1.2 Isopropanol, 80 percent. Mix 80 ml of isopropanol with 20 ml of deionized, distilled water. Check each lot of isopropanol for peroxide impurities as follows: shake 10 ml of isopropanol with 10 ml of freshly prepared 10 percent potassium iodide solution. Prepare a blank by similarly treating 10 ml of distilled water. After 1 minute, read the absorbance at 352 nanometers on a spectrophotometer. If absorbance exceeds 0.1, reject alcohol for use.

Peroxides may be removed from isopropanol by redistilling or by passage through a column of activated alumina; however, reagent grade isopropanol with suitably low peroxide levels may be obtained from commercial sources. Rejection of contaminated lots may, therefore, be a more efficient procedure.

3.1.3 Hydrogen Peroxide, 3 Percent. Dilute 30 percent hydrogen peroxide 1:9 (v/v) with deionized, distilled water (30 ml is needed per sample). Prepare fresh daily.

3.1.4 Potassium Iodide Solution, 10 Percent. Dissolve 10.0 grams KI in deionized, distilled water and dilute to 100 ml. Prepare when needed.

3.2 Sample Recovery.

3.2.1 Water. Deionized, distilled, as in 3.1.1.

3.2.2 Isopropanol, 80 Percent. Mix 80 ml of isopropanol with 20 ml of deionized, distilled water.

3.3 Analysis.

3.3.1 Water. Deionized, distilled, as in 3.1.1.

3.3.2 Isopropanol, 100 percent.

3.3.3 Thorin Indicator. 1-(o-arsonophenylazo)-2-naphthol-3,6-disulfonic acid, disodium salt, or equivalent. Dissolve 0.20 g in 100 ml of deionized, distilled water.

3.3.4 Barium Perchlorate Solution, 0.0100 N. Dissolve 1.95 g of barium perchlorate trihydrate [$Ba(ClO_4)_2 \cdot 3H_2O$] in 200 ml distilled water and dilute to 1 liter with isopropanol. Alternatively, 1.22 g of [$BaCl_2 \cdot 2H_2O$] may be used instead of the perchlorate. Standardize as in Section 5.5.

3.3.5 Sulfuric Acid Standard, 0.0100 N. Purchase or standardize to ±0.0002 N against 0.0100 N NaOH which has previously been standardized against potassium acid phthalate (primary standard grade).

## 4. *Procedure*

4.1 Sampling.

4.1.1 Preparation of collection train. Measure 15 ml of 80 percent isopropanol into the midget bubbler and 15 ml of 3 percent hydrogen peroxide into each of the first two midget impingers. Leave the final midget impinger dry. Assemble the train as shown in Figure 6-1. Adjust probe heater to a temperature sufficient to prevent water condensation. Place crushed ice and water around the impingers.

4.1.2 Leak-check procedure. A leak check prior to the sampling run is optional; however, a leak check after the sampling run is mandatory. The leak-check procedure is as follows:

With the probe disconnected, place a vacuum gauge at the inlet to the bubbler and pull a vacuum of 250 mm (10 in.) Hg; plug or pinch off the outlet of the flow meter, and then turn off the pump. The vacuum shall remain stable for at least 30 seconds. Carefully release the vacuum gauge before releasing the flow meter end to prevent back flow of the impinger fluid.

Other leak check procedures may be used, subject to the approval of the Administrator, U.S. Environmental Protection Agency. The procedure used in Method 5 is not suitable for diaphragm pumps.

4.1.3 Sample collection. Record the initial dry gas meter reading and barometric pressure. To begin sampling, position the tip of the probe at the sampling point, connect the probe to the bubbler, and start the pump. Adjust the sample flow to a constant rate of approximately 1.0 liter/min as indicated by the rotameter. Maintain this constant rate (±10 percent) during the entire sampling run. Take readings (dry gas meter, temperatures at dry gas meter and at impinger outlet and rate meter) at least every 5 minutes. Add more ice during the run to keep the temperature of the gases leaving the last impinger at 20° C (68° F) or less. At the conclusion of each run, turn off the pump, remove probe from the stack, and record the final readings. Conduct a leak check as in Section 4.1.2. (This leak check is mandatory.) If a leak is found, void the test run. Drain the ice bath, and purge the remaining part of the train by drawing clean ambient air through the system for 15 minutes at the sampling rate.

Clean ambient air can be provided by passing air through a charcoal filter or through an extra midget impinger with 15 ml of 3 percent $H_2O_2$. The tester may opt to simply use ambient air, without purification.

4.2 Sample Recovery. Disconnect the impingers after purging. Discard the contents of the midget bubbler. Pour the contents of the midget impingers into a leak-free polyethylene bottle for shipment. Rinse the three midget impingers and the connecting tubes with deionized, distilled water, and add the washings to the same storage container. Mark the fluid level. Seal and identify the sample container.

4.3 Sample Analysis. Note level of liquid in container, and confirm whether any sample was lost during shipment; note this on analytical data sheet. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results.

Transfer the contents of the storage container to a 100-ml volumetric flask and dilute to exactly 100 ml with deionized, distilled water. Pipette a 20-ml aliquot of this solution into a 250-ml [illegible]meyer flask, add 80 ml of 100 percent isopropanol and two to four drops of thorin indicator, and titrate to a pink endpoint using 0.0100 N barium perchlorate. Repeat and average the titration volumes. Run a blank with each series of samples. Replicate titrations must agree within 1 percent or 0.2 ml, whichever is larger.

(NOTE.—Protect the 0.0100 N barium perchlorate solution from evaporation at all times.)

## 5. *Calibration*

5.1 Metering System.

5.1.1 Initial Calibration. Before its initial use in the field, first leak check the metering system (drying tube, needle valve, pump, rotameter, and dry gas meter) as follows: place a vacuum gauge at the inlet to the drying tube and pull a vacuum of 250 mm (10 in.) Hg; plug or pinch off the outlet of the flow meter, and then turn off the pump. The vacuum shall remain stable for at least 30 seconds. Carefully release the vacuum gauge before releasing the flow meter end.

Next, calibrate the metering system (at the sampling flow rate specified by the method) as follows: connect an appropriately sized wet test meter (e.g., 1 liter per revolution) to the inlet of the drying tube. Make three independent calibration runs, using at least five revolutions of the dry gas meter per run. Calculate the calibration factor, Y (wet test meter calibration volume divided by the dry gas meter volume, both volumes adjusted to the same reference temperature and pressure), for each run, and average the results. If any Y value deviates by more than 2 percent from the average, the metering system is unacceptable for use. Otherwise, use the average as the calibration factor for subsequent test runs.

5.1.2 Post-Test Calibration Check. After each field test series, conduct a calibration check as in Section 5.1.1 above, except for the following variations: (a) the leak check is not to be conducted, (b) three, or more revolutions of the dry gas meter may be used, and (c) only two independent runs need be made. If the calibration factor does not deviate by more than 5 percent from the initial calibration factor (determined in Section 5.1.1), then the dry gas meter volumes obtained during the test series are acceptable. If the calibration factor deviates by more than 5 percent, recalibrate the metering system as in Section 5.1.1, and for the calculations, use the calibration factor (initial or recalibration) that yields the lower gas volume for each test run.

5.2 Thermometers. Calibrate against mercury-in-glass thermometers.

5.3 Rotameter. The rotameter need not be calibrated but should be cleaned and maintained according to the manufacturer's instruction.

5.4 Barometer. Calibrate against a mercury barometer.

5.5 Barium Perchlorate Solution. Standardize the barium perchlorate solution against 25 ml of standard sulfuric acid to which 100 ml of 100 percent isopropanol has been added.

## 6. *Calculations*

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

6.1 Nomenclature.

$C_{SO_2}$ = Concentration of sulfur dioxide, dry basis corrected to standard conditions, mg/dscm (lb/dscf).
$N$ = Normality of barium perchlorate titrant, milliequivalents/ml.
$P_{bar}$ = Barometric pressure at the exit orifice of the dry gas meter, mm Hg (in. Hg).
$P_{std}$ = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).
$T_m$ = Average dry gas meter absolute temperature, °K (°R).
$T_{std}$ = Standard absolute temperature, 293° K (528° R).
$V_a$ = Volume of sample aliquot titrated, ml.
$V_m$ = Dry gas volume as measured by the dry gas meter, dcm (dcf).
$V_{m(std)}$ = Dry gas volume measured by the dry gas meter, corrected to standard conditions, dscm (dscf).
$V_{soln}$ = Total volume of solution in which the sulfur dioxide sample is contained, 100 ml.
$V_t$ = Volume of barium perchlorate titrant used for the sample, ml (average of replicate titrations).
$V_{tb}$ = Volume of barium perchlorate titrant used for the blank, ml.
$Y$ = Dry gas meter calibration factor.
32.03 = Equivalent weight of sulfur dioxide.

6.2 Dry sample gas volume, corrected to standard conditions.

$$V_{m(std)} = V_m Y \left(\frac{T_{std}}{T_m}\right)\left(\frac{P_{bar}}{P_{std}}\right) = K_1 Y \frac{V_m P_{bar}}{T_m}$$

Equation 6-1

where:

$K_1$ = 0.3858 °K/mm Hg for metric units.
= 17.64 °R/in. Hg for English units.

6.3 Sulfur dioxide concentration.

$$C_{SO_2} = K_2 \frac{(V_t - V_{tb})\, N \left(\dfrac{V_{soln}}{V_a}\right)}{V_{m(std)}}$$

Equation 6-2

where:

$K_2$ = 32.03 mg/meq. for metric units.
= $7.061 \times 10^{-5}$ lb/meq. for English units.

## 7. *Bibliography*

1. Atmospheric Emissions from Sulfuric Acid Manufacturing Processes. U.S. DHEW, PHS, Division of Air Pollution. Public Health Service Publication No. 999-AP-13. Cincinnati, Ohio. 1965.

2. Corbett, P. F. The Determination of $SO_2$ and $SO_3$ in Flue Gases. Journal of the Institute of Fuel. 24: 237-243, 1961.

3. Matty, R. E. and E. K. Diehl. Measuring Flue-Gas $SO_2$ and $SO_3$. Power. *101*: 94-97. November 1957.

4. Patton, W. F. and J. A. Brink, Jr. New Equipment and Techniques for Sampling Chemical Process Gases. J. Air Pollution Control Association. *13*: 162. 1963.

5. Rom, J. J. Maintenance, Calibration, and Operation of Isokinetic Source-Sampling Equipment. Office of Air Programs, Environmental Protection Agency. Research Triangle Park, N.C. APTD-0576. March 1972.

6. Hamil, H. F. and D. E. Camann. Collaborative Study of Method for the Determination of Sulfur Dioxide Emissions from Stationary Sources (Fossil-Fuel Fired Steam Generators). Environmental Protection Agency, Research Triangle Park, N.C. EPA-650/4-74-024. December 1973.

7. Annual Book of ASTM Standards. Part 31; Water, Atmospheric Analysis. American Society for Testing and Materials. Philadelphia, Pa. 1974. pp. 40-42

8. Knoll, J. E. and M. R. Midgett. The Application of EPA Method 6 to High Sulfur Dioxide Concentrations. Environmental Protection Agency. Research Triangle Park, N.C. EPA-600/4-76-038. July 1976.

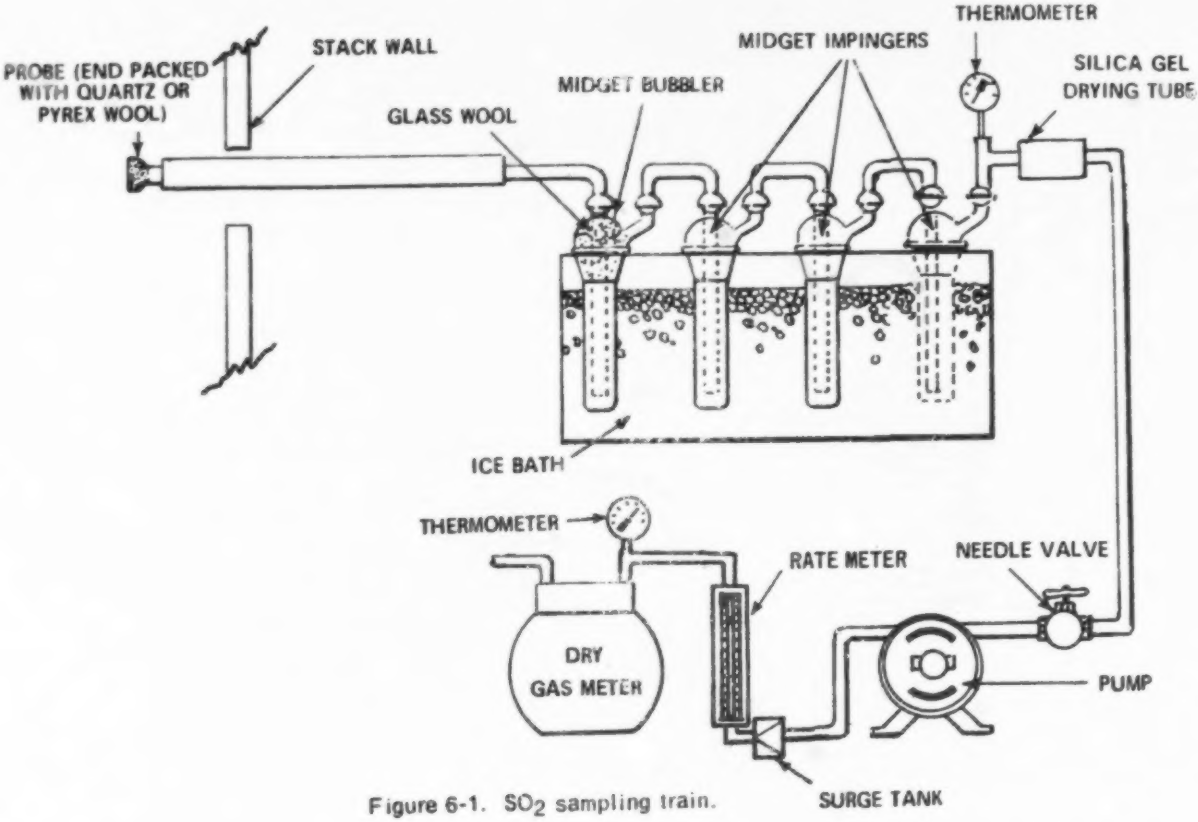

PROBE (END PACKED WITH QUARTZ OR PYREX WOOL)
STACK WALL
GLASS WOOL
MIDGET BUBBLER
MIDGET IMPINGERS
THERMOMETER
SILICA GEL DRYING TUBE
ICE BATH
THERMOMETER
RATE METER
NEEDLE VALVE
DRY GAS METER
PUMP
SURGE TANK

Figure 6-1. $SO_2$ sampling train.

# METHOD 7

# DETERMINATION OF NITROGEN OXIDE EMISSIONS FROM STATIONARY SOURCES

## 1. *Principle and Applicability*

1.1 Principle. A grab sample is collected in an evacuated flask containing a dilute sulfuric acid-hydrogen peroxide absorbing solution, and the nitrogen oxides, except nitrous oxide, are measured colorimeterically using the phenoldisulfonic acid (PDS) procedure.

1.2 Applicability. This method is applicable to the measurement of nitrogen oxides emitted from stationary sources. The range of the method has been determined to be 2 to 400 milligrams $NO_x$ (as $NO_2$) per dry standard cubic meter, without having to dilute the sample.

## 2. *Apparatus*

2.1 Sampling (see Figure 7-1). Other grab sampling systems or equipment, capable of measuring sample volume to within ±2.0 percent and collecting a sufficient sample volume to allow analytical reproducibility to within ±5 percent, will be considered acceptable alternatives, subject to approval of the Administrator, U.S. Environmental Protection Agency. The following equipment is used in sampling:

2.1.1 Probe. Borosilicate glass tubing, sufficiently heated to prevent water condensation and equipped with an in-stack or out-stack filter to remove particulate matter (a plug of glass wool is satisfactory for this purpose). Stainless steel or Teflon[3] tubing may also be used for the probe. Heating is not necessary if the probe remains dry during the purging period.

2.1.2 Collection Flask. Two-liter borosilicate, round bottom flask, with short neck and 24/40 standard taper opening, protected against implosion or breakage.

2.1.3 Flask Valve. T-bore stopcock connected to a 24/40 standard taper joint.

2.1.4 Temperature Gauge. Dial-type thermometer, or other temperature gauge, capable of measuring 1° C (2° F) intervals from −5 to 50° C (25 to 125° F).

2.1.5 Vacuum Line. Tubing capable of withstanding a vacuum of 75 mm Hg (3 in. Hg) absolute pressure, with "T" connection and T-bore stopcock.

2.1.6 Vacuum Gauge. U-tube manometer, 1 meter (36 in.), with 1-mm (0.1-in.) divisions, or other gauge capable of measuring pressure to within ±2.5 mm Hg (0.10 in. Hg).

2.1.7 Pump. Capable of evacuating the collection flask to a pressure equal to or less than 75 mm Hg (3 in. Hg) absolute.

2.1.8 Squeeze Bulb. One-way.

2.1.9 Volumetric Pipette. 25 ml.

2.1.10 Stopcock and Ground Joint Grease. A high-vacuum, high-temperature chlorofluorocarbon grease is required. Halocarbon 25-5S has been found to be effective.

2.1.11 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby national weather service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase, or vice versa for elevation decrease.

2.2 Sample Recovery. The following equipment is required for sample recovery:

2.2.1 Graduated Cylinder. 50 ml with 1-ml divisions.

2.2.2 Storage Containers. Leak-free polyethylene bottles.

2.2.3 Wash Bottle. Polyethylene or glass.

2.2.4 Glass Stirring Rod.

2.2.5 Test Paper for Indicating pH. To cover the pH range of 7 to 14.

2.3 Analysis. For the analysis, the following equipment is needed:

2.3.1 Volumetric Pipettes. Two 1 ml, two 2 ml, one 3 ml, one 4 ml, two 10 ml, and one 25 ml for each sample and standard.

2.3.2 Porcelain Evaporating Dishes. 175- to 250-ml capacity with lip for pouring, one for each sample and each standard. The Coors No. 45066 (shallow-form, 195 ml) has been found to be satisfactory. Alternatively, polymethyl pentene beakers (Nalge No. 1203, 150 ml), or glass beakers (150 ml) may be used. When glass beakers are used, etching of the beakers may cause solid matter to be present in the analytical step; the solids should be removed by filtration (see Section 4.3).

2.3.3 Steam Bath. Low-temperature ovens or thermostatically controlled hot plates kept below 70° C (160° F) are acceptable alternatives.

2.3.4 Dropping Pipette or Dropper. Three required.

2.3.5 Polyethylene Policeman. One for each sample and each standard.

2.3.6 Graduated Cylinder. 100 ml with 1-ml divisions.

2.3.7 Volumetric Flasks. 50 ml (one for each sample), 100 ml (one for each sample and each standard, and one for the working standard $KNO_3$ solution), and 1000 ml (one).

2.3.8 Spectrophotometer. To measure absorbance at 410 nm.

2.3.9 Graduated Pipette. 10 ml with 0.1-ml divisions.

2.3.10 Test Paper for Indicating pH. To cover the pH range of 7 to 14.

2.3.11 Analytical Balance. To measure to within 0.1 mg.

## 3. *Reagents*

Unless otherwise indicated, it is intended that all reagents conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available; otherwise, use the best available grade.

3.1 Sampling. To prepare the absorbing solution, cautiously add 2.8 ml concentrated $H_2SO_4$ to 1 liter of deionized, distilled water. Mix well and add 6 ml of 3 percent hydrogen peroxide, freshly prepared from 30 percent hydrogen peroxide solution. The absorbing solution should be used within 1 week of its preparation. Do not expose to extreme heat or direct sunlight.

3.2 Sample Recovery. Two reagents are required for sample recovery:

3.2.1 Sodium Hydroxide (1N). Dissolve 40 g NaOH in deionized, distilled water and dilute to 1 liter.

3.2.2 Water. Deionized, distilled to conform to ASTM specification D1193-74, Type 3. At the option of the analyst, the $KMNO_4$ test for oxidizable organic matter may be omitted when high concentrations of organic matter are not expected to be present.

3.3 Analysis. For the analysis, the following reagents are required:

3.3.1 Fuming Sulfuric Acid. 15 to 18 percent by weight free sulfur trioxide. HANDLE WITH CAUTION.

3.3.2 Phenol. White solid.

3.3.3 Sulfuric Acid. Concentrated, 95 percent minimum assay. HANDLE WITH CAUTION.

3.3.4 Potassium Nitrate. Dried at 105 to 110° C (220 to 230° F) for a minimum of 2 hours just prior to preparation of standard solution.

3.3.5 Standard $KNO_3$ Solution. Dissolve exactly 2.198 g of dried potassium nitrate ($KNO_3$) in deionized, distilled water and dilute to 1 liter with deionized, distilled water in a 1,000-ml volumetric flask.

3.3.6 Working Standard $KNO_3$ Solution. Dilute 10 ml of the standard solution to 100 ml with deionized distilled water. One milliliter of the working standard solution is equivalent to 100 μg nitrogen dioxide ($NO_2$).

3.3.7 Water. Deionized, distilled as in Section 3.2.2.

3.3.8 Phenoldisulfonic Acid Solution. Dissolve 25 g of pure white phenol in 150 ml concentrated sulfuric acid on a steam bath. Cool, add 75 ml fuming sulfuric acid, and heat at 100° C (212° F) for 2 hours. Store in a dark, stoppered bottle.

## 4. *Procedures*

4.1 Sampling.

4.1.1 Pipette 25 ml of absorbing solution into a sample flask, retaining a sufficient quantity for use in preparing the calibration standards. Insert the flask valve stopper into the flask with the valve in the "purge" position. Assemble the sampling train as shown in Figure 7-1 and place the probe at the sampling point. Make sure that all fittings are tight and leak-free, and that all ground glass joints have been properly greased with a high-vacuum, high-temperature chlorofluorocarbon-based stopcock grease. Turn the flask valve and the pump valve to their "evacuate" positions. Evacuate the flask to 75 mm Hg (3 in. Hg) absolute pressure, or less. Evacuation to a pressure approaching the vapor pressure of water at the existing temperature is desirable. Turn the pump valve to its "vent" position and turn off the pump. Check for leakage by observing the manometer for any pressure fluctuation. (Any variation greater than 10 mm Hg (0.4 in. Hg) over a period of 1 minute is not acceptable, and the flask is not to be used until the leakage problem is corrected. Pressure in the flask is not to exceed 75 mm Hg (3 in. Hg) absolute at the time sampling is commenced.) Record the volume of the flask and valve ($V_f$), the flask temperature ($T_i$), and the barometric pressure. Turn the flask valve counterclockwise to its "purge" position and do the same with the pump valve. Purge the probe and the vacuum tube using the squeeze bulb. If condensation occurs in the probe and the flask valve area, heat the probe and purge until the condensation disappears. Next, turn the pump valve to its "vent" position. Turn the flask valve clockwise to its "evacuate" position and record the difference in the mercury levels in the manometer. The absolute internal pressure in the flask ($P_i$) is equal to the barometric pressure less the manometer reading. Immediately turn the flask valve to the "sample" position and permit the gas to enter the flask until pressures in the flask and sample line (i.e., duct, stack) are equal. This will usually require about 15 seconds; a longer period indicates a "plug" in the probe, which must be corrected before sampling is continued. After collecting the sample, turn the flask valve to its "purge" position and disconnect the flask from the sampling train. Shake the flask for at least 5 minutes.

---

[3] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

4.1.2 If the gas being sampled contains insufficient oxygen for the conversion of NO to $NO_2$ (e.g., an applicable subpart of the standard may require taking a sample of a calibration gas mixture of NO in $N_2$), then oxygen shall be introduced into the flask to permit this conversion. Oxygen may be introduced into the flask by one of three methods; (1) Before evacuating the sampling flask, flush with pure cylinder oxygen, then evacuate flask to 75 mm Hg (3 in. Hg) absolute pressure or less; or (2) inject oxygen into the flask after sampling; or (3) terminate sampling with a minimum of 50 mm Hg (2 in. Hg) vacuum remaining in the flask, record this final pressure, and then vent the flask to the atmosphere until the flask pressure is almost equal to atmospheric pressure.

4.2 Sample Recovery. Let the flask set for a minimum of 16 hours and then shake the contents for 2 minutes. Connect the flask to a mercury filled U-tube manometer. Open the valve from the flask to the manometer and record the flask temperature ($T_f$), the barometric pressure, and the difference between the mercury levels in the manometer. The absolute internal pressure in the flask ($P_f$) is the barometric pressure less the manometer reading. Transfer the contents of the flask to a leak-free polyethylene bottle. Rinse the flask twice with 5-ml portions of deionized, distilled water and add the rinse water to the bottle. Adjust the pH to between 9 and 12 by adding sodium hydroxide (1 N), dropwise (about 25 to 35 drops). Check the pH by dipping a stirring rod into the solution and then touching the rod to the pH test paper. Remove as little material as possible during this step. Mark the height of the liquid level so that the container can be checked for leakage after transport. Label the container to clearly identify its contents. Seal the container for shipping.

4.3 Analysis. Note the level of the liquid in container and confirm whether or not any sample was lost during shipment; note this on the analytical data sheet. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Immediately prior to analysis, transfer the contents of the shipping container to a 50-ml volumetric flask, and rinse the container twice with 5-ml portions of deionized, distilled water. Add the rinse water to the flask and dilute to the mark with deionized, distilled water; mix thoroughly. Pipette a 25-ml aliquot into the procelain evaporating dish. Return any unused portion of the sample to the polyethylene storage bottle. Evaporate the 25-ml aliquot to dryness on a steam bath and allow to cool. Add 2 ml phenoldisulfonic acid solution to the dried residue and triturate thoroughly with a poylethylene policeman. Make sure the solution contacts all the residue. Add 1 ml deionized, distilled water and four drops of concentrated sulfuric acid. Heat the solution on a steam bath for 3 minutes with occasional stirring. Allow the solution to cool, add 20 ml deionized, distilled water, mix well by stirring, and add concentrated ammonium hydroxide, dropwise, with constant stirring, until the pH is 10 (as determined by pH paper). If the sample contains solids, these must be removed by filtration (centrifugation is an acceptable alternative, subject to the approval of the Administrator), as follows: filter through Whatman No. 41 filter paper into a 100-ml volumetric flask; rinse the evaporating dish with three 5-ml portions of deionized, distilled water; filter these three rinses. Wash the filter with at least three 15-ml portions of deionized, distilled water. Add the filter washings to the contents of the volumetric flask and dilute to the mark with deionized, distilled water. If solids are absent, the solution can be transferred directly to the 100-ml volumetric flask and diluted to the mark with deionized, distilled water. Mix the contents of the flask thoroughly, and measure the absorbance at the optimum wavelength used for the standards (Section 5.2.1), using the blank solution as a zero reference. Dilute the sample and the blank with equal volumes of deionized, distilled water if the absorbance exceeds $A_4$, the absorbance of the 400 μg $NO_2$ standard (see Section 5.2.2).

### 5. *Calibration*

5.1 Flask Volume. The volume of the collection flask-flask valve combination must be known prior to sampling. Assemble the flask and flask valve and fill with water, to the stopcock. Measure the volume of water to ±10 ml. Record this volume on the flask.

5.2 Spectrophotometer Calibration.

5.2.1 Optimum Wavelength Determination. For both fixed and variable wavelength spectrophotometers, calibrate against standard certified wavelength of 410 nm, every 6 months. Alternatively, for variable wave length spectrophotometers, scan the spectrum between 400 and 415 nm using a 200 μg $NO_2$ standard solution (see Section 5.2.2). If a peak does not occur, the spectrophotometer is probably malfunctioning, and should be repaired. When a peak is obtained within the 400 to 415 nm range, the wavelength at which this peak occurs shall be the optimum wavelength for the measurement of absorbance for both the standards and samples.

5.2.2 Determination of Spectrophotometer Calibration Factor $K_c$. Add 0.0, 1.0, 2.0, 3.0, and 4.0 ml of the $KNO_3$ working standard solution (1 ml=100 μg $NO_2$) to a series of five porcelain evaporating dishes. To each, add 25 ml of absorbing solution, 10 ml deionized, distilled water, and sodium hydroxide (1N), dropwise, until the pH is between 9 and 12 (about 25 to 35 drops each). Beginning with the evaporation step, follow the analysis procedure of Section 4.3, until the solution has been transferred to the 100 ml volumetric flask and diluted to the mark. Measure the absorbance of each solution, at the optimum wavelength, as determined in Section 5.2.1. This calibration procedure must be repeated on each day that samples are analyzed. Calculate the spectrophotometer calibration factor as follows:

$$K_c = 100\,\frac{A_1 + 2A_2 + 3A_3 + 4A_4}{A_1^2 + A_2^2 + A_3^2 + A_4^2}$$

Equation 7–1

where:

$K_c$=Calibration factor
$A_1$=Absorbance of the 100-μg $NO_2$ standard
$A_2$=Absorbance of the 200-μg $NO_2$ standard
$A_3$=Absorbance of the 300-μg $NO_2$ standard
$A_4$=Absorbance of the 400-μg $NO_2$ standard

5.3 Barometer. Calibrate against a mercury barometer.

5.4 Temperature Gauge. Calibrate dial thermometers against mercury-in-glass thermometers.

5.5 Vacuum Gauge. Calibrate mechanical gauges, if used, against a mercury manometer such as that specified in 2.1.6.

5.6 Analytical Balance. Calibrate against standard weights.

### 6. *Calculations*

Carry out the calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculations.

6.1 Nomenclature.

$A$=Absorbance of sample.
$C$=Concentration of $NO_x$ as $NO_2$, dry basis, corrected to standard conditions, mg/dscm (lb/dscf).
$F$=Dilution factor (i.e., 25/5, 25/10, etc., required only if sample dilution was needed to reduce the absorbance into the range of calibration).
$K_c$=Spectrophotometer calibration factor.
$m$=Mass of $NO_x$ as $NO_2$ in gas sample, μg.
$P_f$=Final absolute pressure of flask, mm Hg (in. Hg).
$P_i$=Initial absolute pressure of flask, mm Hg (in. Hg).
$P_{std}$=Standard absolute pressure, 760 mm Hg (29.92 in Hg).
$T_f$=Final absolute temperature of flask, °K (°R).
$T_i$=Initial absolute temperature of flask, °K (°R).
$T_{std}$=Standard absolute temperature, 293° K (528° R)
$V_{sc}$=Sample volume at standard conditions (dry basis), ml.
$V_f$=Volume of flask and valve, ml.
$V_a$=Volume of absorbing solution, 25 ml.
2=50/25, the aliquot factor. (If other than a 25-ml aliquot was used for analysis, the corresponding factor must be substituted).

6.2 Sample volume, dry basis, corrected to standard conditions.

$$V_{sc} = \frac{T_{std}}{P_{std}}\,(V_f - V_a)\left[\frac{P_f}{T_f} - \frac{P_i}{T_i}\right]$$

$$= K_1(V_f - 25\ \text{ml})\left[\frac{P_f}{T_f} - \frac{P_i}{T_i}\right]$$

Equation 7–2

where:

$$K_1 = 0.3858\ \frac{°\text{K}}{\text{mm Hg}}\ \text{for metric units}$$

$$= 17.64\ \frac{°\text{R}}{\text{in. Hg}}\ \text{for English units}$$

6.3 Total $\mu g$ $NO_2$ per sample.

$$m=2\,K_c\,AF$$

Equation 7–3

NOTE.—If other than a 25-ml aliquot is used for analysis, the factor 2 must be replaced by a corresponding factor.

6.4 Sample concentration, dry basis, corrected to standard conditions.

$$C=K_2\frac{m}{V_{sc}}$$

Equation 7–4

where:

$$K_2=10^3\ \frac{\text{mg/m}^3}{\mu\text{g/ml}}\ \text{for metric units}$$

$$=6.243\times10^{-5}\ \frac{\text{lb/scf}}{\mu\text{g/ml}}\ \text{for English units}$$

7. *Bibliography*

1. Standard Methods of Chemical Analysis. 6th ed. New York, D. Vua Nostrand Co., Inc. 1962. Vol. 1, p. 325–330.

2. Standard Method of Test for Oxides of Nitrogen in Gaseous Combustion Products (Phenoldisulfonic Acid Procedure). In: 1968 Book of ASTM Standards, Part 26. Philadelphia, Pa. 1968. ASTM Designation D–1608–60, p. 725–729.

3. Jacob, M. B. The Chemical Analysis of Air Pollutants. New York. Interscience Publishers, Inc. 1960. Vol. 10, p. 351–356.

4. Beatty, R. L., L. B. Berger, and H. H. Schrenk. Determination of Oxides of Nitrogen by the Phenoldisulfonic Acid Method. Bureau of Mines, U.S. Dept. of Interior. R. I. 3687. February 1943.

5. Hamil, H. F. and D. E. Camann. Collaborative Study of Method for the Determination of Nitrogen Oxide Emissions from Stationary Sources (Fossil Fuel-Fired Steam Generators). Southwest Research Institute report for Environmental Protection Agency. Research Triangle Park, N.C. October 5, 1973.

6. Hamil, H. F. and R. E. Thomas. Collaborative Study of Method for the Drtermination of Nitrogen Oxide Emissions from Stationary Sources (Nitric Acid Plants). Southwest Research Institute report for Environmental Protection Agency. Research Triangle Park, N.C. May 8, 1974.

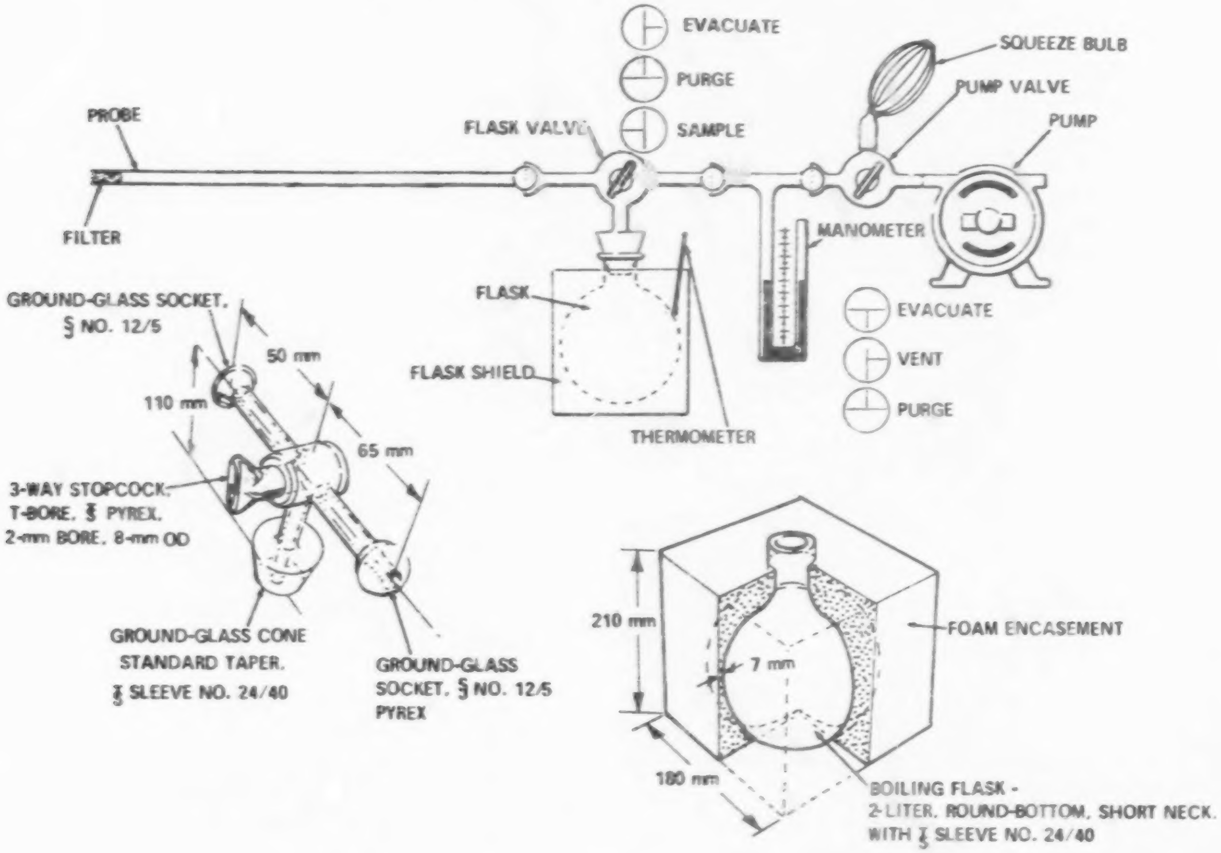

EVACUATE
PURGE
SAMPLE
SQUEEZE BULB
PUMP VALVE
PUMP
PROBE
FLASK VALVE
FILTER
MANOMETER
FLASK
FLASK SHIELD
THERMOMETER
EVACUATE
VENT
PURGE
GROUND-GLASS SOCKET, § NO. 12/5
50 mm
110 mm
65 mm
3-WAY STOPCOCK; T-BORE, § PYREX, 2-mm BORE, 8-mm OD
GROUND-GLASS CONE STANDARD TAPER, § SLEEVE NO. 24/40
GROUND-GLASS SOCKET, § NO. 12/5 PYREX
210 mm
7 mm
180 mm
FOAM ENCASEMENT
BOILING FLASK - 2-LITER, ROUND-BOTTOM, SHORT NECK, WITH § SLEEVE NO. 24/40

Figure 7-1. Sampling train, flask valve, and flask.

# METHOD 8

## DETERMINATION OF SULFURIC ACID MIST AND SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES

### 1. *Principle and Applicability*

1.1 Principle. A gas sample is extracted isokinetically from the stack. The sulfuric acid mist (including sulfur trioxide) and the sulfur dioxide are separated, and both fractions are measured separately by the barium-thorin titration method.

1.2 Applicability. This method is applicable for the determination of sulfuric acid mist (including sulfur trioxide, and in the absence of other particulate matter) and sulfur dioxide emissions from stationary sources. Collaborative tests have shown that the minimum detectable limits of the method are 0.05 milligrams/cubic meter ($0.03\times10^{-7}$ pounds/cubic foot) for sulfur trioxide and 1.2 mg/m³ ($0.74\times10^{-7}$ lb/ft³) for sulfur dioxide. No upper limits have been established. Based on theoretical calculations for 200 milliliters of 3 percent hydrogen peroxide solution, the upper concentration limit for sulfur dioxide in a 1.0 m³ (35.3 ft³) gas sample is about 12,500 mg/m³ ($7.7\times10^{-4}$ lb/ft³). The upper limit can be extended by increasing the quantity of peroxide solution in the impingers.

Possible interfering agents of this method are fluorides, free ammonia, and dimethyl aniline. If any of these interfering agents are present (this can be determined by knowledge of the process), alternative methods, subject to the approval of the Administrator, are required.

Filterable particulate matter may be determined along with $SO_3$ and $SO_2$ (subject to the approval of the Administrator); however, the procedure used for particulate matter must be consistent with the specifications and procedures given in Method 5.

### 2. *Apparatus*

2.1 Sampling. A schematic of the sampling train used in this method is shown in Figure 8-1; it is similar to the Method 5 train except that the filter position is different and the filter holder does not have to be heated. Commercial models of this train are available. For those who desire to build their own, however, complete construction details are described in APTD-0581. Changes from the APTD-0581 document and allowable modifications to Figure 8-1 are discussed in the following subsections.

The operating and maintenance procedures for the sampling train are described in APTD-0576. Since correct usage is important in obtaining valid results, all users should read the APTD-0576 document and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein. Further details and guidelines on operation and maintenance are given in Method 5 and should be read and followed whenever they are applicable.

2.1.1 Probe Nozzle. Same as Method 5, Section 2.1.1.

2.1.2 Probe Liner. Borosilicate or quartz glass, with a heating system to prevent visible condensation during sampling. Do not use metal probe liners.

2.1.3 Pitot Tube. Same as Method 5, Section 2.1.3.

2.1.4 Differential Pressure Gauge. Same as Method 5, Section 2.1.4.

2.1.5 Filter Holder. Borosilicate glass, with a glass frit filter support and a silicone rubber gasket. Other gasket materials, e.g., Teflon or Viton, may be used subject to the approval of the Administrator. The holder design shall provide a positive seal against leakage from the outside or around the filter. The filter holder shall be placed between the first and second impingers. Note: Do not heat the filter holder.

2.1.6 Impingers—Four, as shown in Figure 8-1. The first and third shall be of the Greenburg-Smith design with standard tips. The second and fourth shall be of the Greenburg-Smith design, modified by replacing the insert with an approximately 13 millimeter (0.5 in.) ID glass tube, having an unconstricted tip located 13 mm (0.5 in.) from the bottom of the flask. Similar collection systems, which have been approved by the Administrator, may be used.

2.1.7 Metering System. Same as Method 5, Section 2.1.8.

2.1.8 Barometer. Same as Method 5, Section 2.1.9.

2.1.9 Gas Density Determination Equipment. Same as Method 5, Section 2.1.10.

2.1.10 Temperature Gauge. Thermometer, or equivalent, to measure the temperature of the gas leaving the impinger train to within 1° C (2° F).

2.2 Sample Recovery.

2.2.1 Wash Bottles. Polyethylene or glass, 500 ml. (two).

2.2.2 Graduated Cylinders. 250 ml, 1 liter. (Volumetric flasks may also be used.)

2.2.3 Storage Bottles. Leak-free polyethylene bottles, 1000 ml size (two for each sampling run).

2.2.4 Trip Balance. 500-gram capacity, to measure to ±0.5 g (necessary only if a moisture content analysis is to be done).

2.3 Analysis.

2.3.1 Pipettes. Volumetric 25 ml, 100 ml.

2.3.2 Burrette. 50 ml.

2.3.3 Erlenmeyer Flask. 250 ml. (one for each sample blank and standard).

2.3.4 Graduated Cylinder. 100 ml.

2.3.5 Trip Balance. 500 g capacity, to measure to ±0.5 g.

2.3.6 Dropping Bottle. To add indicator solution, 125-ml size.

### 3. *Reagents*

Unless otherwise indicated, all reagents are to conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Otherwise, use the best available grade.

3.1 Sampling.

3.1.1 Filters. Same as Method 5, Section 3.1.1.

3.1.2 Silica Gel. Same as Method 5, Section 3.1.2.

3.1.3 Water. Deionized, distilled to conform to ASTM specification D1193-74, Type 3. At the option of the analyst, the $KMnO_4$ test for oxidizable organic matter may be omitted when high concentrations of organic matter are not expected to be present.

3.1.4 Isopropanol. 80 Percent. Mix 800 ml of isopropanol with 200 ml of deionized, distilled water.

NOTE.—Experience has shown that only A.C.S. grade isopropanol is satisfactory. Tests have shown that isopropanol obtained from commercial sources occasionally has peroxide impurities that will cause erroneously high sulfuric acid mist measurement. Use the following test for detecting peroxides in each lot of isopropanol: Shake 10 ml of the isopropanol with 10 ml of freshly prepared 10 percent potassium iodide solution. Prepare a blank by similarly treating 10 ml of distilled water. After 1 minute, read the absorbance on a spectrophotometer at 352 nanometers. If the absorbance exceeds 0.1, the isopropanol shall not be used. Peroxides may be removed from isopropanol by redistilling, or by passage through a column of activated alumina. However, reagent-grade isopropanol with suitably low peroxide levels is readily available from commercial sources; therefore, rejection of contaminated lots may be more efficient than following the peroxide removal procedure.

3.1.5 Hydrogen Peroxide, 3 Percent. Dilute 100 ml of 30 percent hydrogen peroxide to 1 liter with deionized, distilled water. Prepare fresh daily.

3.1.6 Crushed Ice.

3.2 Sample Recovery.

3.2.1 Water. Same as 3.1.3.

3.2.2 Isopropanol, 80 Percent. Same as 3.1.4.

3.3 Analysis.

3.3.1 Water. Same as 3.1.3.

3.3.2 Isopropanol, 100 Percent.

3.3.3 Thorin Indicator. 1-(o-arsonophenylazo)-2-naphthol-3, 6-disulfonic acid, disodium salt, or equivalent. Dissolve 0.20 g in 100 ml of deionized, distilled water.

3.3.4 Barium Perchlorate (0.0100 Normal). Dissolve 1.95 g of barium perchlorate trihydrate ($Ba(C10_4)_2 \cdot 3H_2O$) in 200 ml deionized, distilled water, and dilute to 1 liter with isopropanol; 1.22 g of barium chloride dihydrate ($BaCl_2 \cdot 2H_2O$) may be used instead of the barium perchlorate. Standardize with sulfuric acid as in Section 5.2. This solution must be protected against evaporation at all times.

3.3.5 Sulfuric Acid Standard (0.0100 N). Purchase or standardize to ±0.0002 N against 0.0100 N NaOH that has previously been standardized against primary standard potassium acid phthalate.

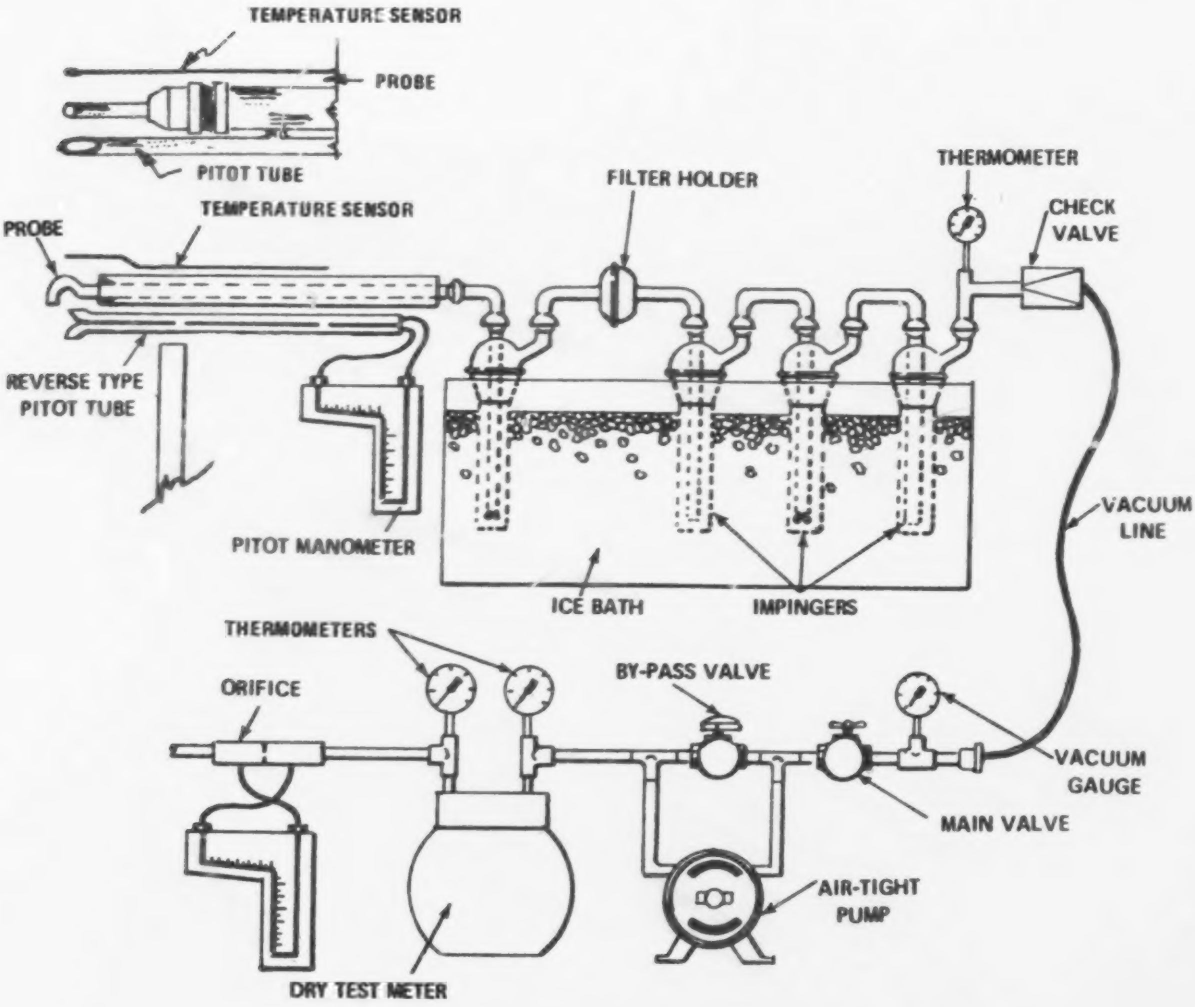

TEMPERATURE SENSOR
PROBE
PITOT TUBE
PROBE
TEMPERATURE SENSOR
REVERSE TYPE PITOT TUBE
PITOT MANOMETER
FILTER HOLDER
THERMOMETER
CHECK VALVE
VACUUM LINE
ICE BATH
IMPINGERS
THERMOMETERS
ORIFICE
BY-PASS VALVE
VACUUM GAUGE
MAIN VALVE
AIR-TIGHT PUMP
DRY TEST METER

Figure 8-1. Sulfuric acid mist sampling train.

## 4. *Procedure*

### 4.1 Sampling.

4.1.1 Pretest Preparation. Follow the procedure outlined in Method 5, Section 4.1.1; filters should be inspected, but need not be desiccated, weighed, or identified. If the effluent gas can be considered dry, i.e., moisture free, the silica gel need not be weighed.

4.1.2 Preliminary Determinations. Follow the procedure outlined in Method 5, Section 4.1.2.

4.1.3 Preparation of Collection Train. Follow the procedure outlined in Method 5, Section 4.1.3 (except for the second paragraph and other obviously inapplicable parts) and use Figure 8–1 instead of Figure 5–1. Replace the second paragraph with: Place 100 ml of 80 percent isopropanol in the first impinger, 100 ml of 3 percent hydrogen peroxide in both the second and third impingers; retain a portion of each reagent for use as a blank solution. Place about 200 g of silica gel in the fourth impinger.

NOTE.—If moisture content is to be determined by impinger analysis, weigh each of the first three impingers (plus absorbing solution) to the nearest 0.5 g and record these weights. The weight of the silica gel (or silica gel plus container) must also be determined to the nearest 0.5 g and recorded.

4.1.4 Pretest Leak-Check Procedure. Follow the basic procedure outlined in Method 5, Section 4.1.4.1, noting that the probe heater shall be adjusted to the minimum temperature required to prevent condensation, and also that verbage such as, "* * * plugging the inlet to the filter holder * * *," shall be replaced by, "* * * plugging the inlet to the first impinger * * *." The pretest leak-check is optional.

4.1.5 Train Operation. Follow the basic procedures outlined in Method 5, Section 4.1.5, in conjunction with the following special instructions. Data shall be recorded on a sheet similar to the one in Figure 8–2. The sampling rate shall not exceed 0.030 m³/min (1.0 cfm) during the run. Periodically during the test, observe the connecting line between the probe and first impinger for signs of condensation. If it does occur, adjust the probe heater setting upward to the minimum temperature required to prevent condensation. If component changes become necessary during a run, a leak-check shall be done immediately before each change, according to the procedure outlined in Section 4.1.4.2 of Method 5 (with appropriate modifications, as mentioned in Section 4.1.4 of this method); record all leak rates. If the leakage rate(s) exceed the specified rate, the tester shall either void the run or shall plan to correct the sample volume as outlined in Section 6.3 of Method 5. Immediately after component changes, leak-checks are optional. If these leak-checks are done, the procedure outlined in Section 4.1.4.1 of Method 5 (with appropriate modifications) shall be used.

After turning off the pump and recording the final readings at the conclusion of each run, remove the probe from the stack. Conduct a post-test (mandatory) leak-check as in Section 4.1.4.3 of Method 5 (with appropriate modification) and record the leak rate. If the post-test leakage rate exceeds the specified acceptable rate, the tester shall either correct the sample volume, as outlined in Section 6.3 of Method 5, or shall void the run.

Drain the ice bath and, with the probe disconnected, purge the remaining part of the train, by drawing clean ambient air through the system for 15 minutes at the average flow rate used for sampling.

NOTE.—Clean ambient air can be provided by passing air through a charcoal filter. At the option of the tester, ambient air (without cleaning) may be used.

4.1.6 Calculation of Percent Isokinetic. Follow the procedure outlined in Method 5, Section 4.1.6.

### 4.2 Sample Recovery.

4.2.1 Container No. 1. If a moisture content analysis is to be done, weigh the first impinger plus contents to the nearest 0.5 g and record this weight.

Transfer the contents of the first impinger to a 250-ml graduated cylinder. Rinse the probe, first impinger, all connecting glassware before the filter, and the front half of the filter holder with 80 percent isopropanol. Add the rinse solution to the cylinder. Dilute to 250 ml with 80 percent isopropanol. Add the filter to the solution, mix, and transfer to the storage container. Protect the solution against evaporation. Mark the level of liquid on het container and identify the sample container.

4.2.2 Container No. 2. If a moisture content analysis is to be done, weigh the second and third impingers (plus contents) to the nearest 0.5 g and record these weights. Also, weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g.

Transfer the solutions from the second and third impingers to a 1000-ml graduated cylinder. Rinse all connecting glassware (including back half of filter holder) between the filter and silica gel impinger with deionized, distilled water, and add this rinse water to the cylinder. Dilute to a volume of 1000 ml with deionized, distilled water. Transfer the solution to a storage container. Mark the level of liquid on the container. Seal and identify the sample container.

### 4.3 Analysis.

Note the level of liquid in containers 1 and 2, and confirm whether or not any sample was lost during shipment; note this on the analytical data sheet. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results.

4.3.1 Container No. 1. Shake the container holding the isopropanol solution and the filter. If the filter breaks up, allow the fragments to settle for a few minutes before removing a sample. Pipette a 100-ml aliquot of this solution into a 250-ml Erlenmeyer flask, add 2 to 4 drops of thorin indicator, and titrate to a pink endpoint using 0.0100 N barium perchlorate. Repeat the titration with a second aliquot of sample and average the titration values. Replicate titrations must agree within 1 percent or 0.2 ml, whichever is greater.

4.3.2 Container No. 2. Thoroughly mix the solution in the container holding the contents of the second and third impingers. Pipette a 10-ml aliquot of sample into a 250-ml Erlenmeyer flask. Add ml of isopropanol, 2 to 4 drops of thorin indicator, and titrate to a pink endpoint using 0.0100 N barium perchlorate. Repeat the titration with a second aliquot of sample and average the titration values. Replicate titrations must agree within 1 percent or 0.2 ml, whichever is greater.

4.3.3 Blanks. Prepare blanks by adding 2 to 4 drops of thorin indicator to 100 ml of 80 percent isopropanol. Titrate the blanks in the same manner as the samples.

## 5. *Calibration*

5.1 Calibrate equipment using the procedures specified in the following sections of Method 5: Section 5.3 (metering system); Section 5.5 (temperature gauges); Section 5.7 (barometer). Note that the recommended leak-check of the metering system, described in Section 5.6 of Method 5, also applies to this method.

5.2 Standardize the barium perchlorate solution with 25 ml of standard sulfuric acid, to which 100 ml of 100 percent isopropanol has been added.

## 6. *Calculations*

Note.—Carry out calculations retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

### 6.1 Nomenclature.

$A_n$ = Cross-sectional area of nozzle, m² (ft²).

$B_{ws}$ = Water vapor in the gas stream, proportion by volume.

$C_{H_2SO_4}$ = Sulfuric acid (including $SO_3$) concentration, g/dscm (lb/dscf).

$C_{SO_2}$ = Sulfur dioxide concentration, g/dscm (lb/dscf).

$I$ = Percent of isokinetic sampling.

$N$ = Normality of barium perchlorate titrant, g equivalents/liter.

$P_{bar}$ = Barometric pressure at the sampling site, mm Hg (in. Hg).

$P_s$ = Absolute stack gas pressure, mm Hg (in. Hg).

$P_{std}$ = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

$T_m$ = Average absolute dry gas meter temperature (see Figure 8–2), ° K (° R).

$T_s$ = Average absolute stack gas temperature (see Figure 8–2), ° K (° R).

$T_{std}$ = Standard absolute temperature, 293° K (528° R).

$V_a$ = Volume of sample aliquot titrated, 100 ml for $H_2SO_4$ and 10 ml for $SO_2$.

$V_{lc}$ = Total volume of liquid collected in impingers and silica gel, ml.

$V_m$ = Volume of gas sample as measured by dry gas meter, dcm (dcf).

$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter corrected to standard conditions, dscm (dscf).

$v_s$ = Average stack gas velocity, calculated by Method 2, Equation 2-9, using data obtained from Method 8, m/sec (ft/sec).

$V_{soln}$ = Total volume of solution in which the sulfuric acid or sulfur dioxide sample is contained, 250 ml or 1,000 ml, respectively.

$V_t$ = Volume of barium perchlorate titrant used for the sample, ml.

$V_{tb}$ = Volume of barium perchlorate titrant used for the blank, ml.

$Y$ = Dry gas meter calibration factor.

$\Delta H$ = Average pressure drop across orifice meter, mm (in.) $H_2O$.

$\Theta$ = Total sampling time, min.

13.6 = Specific gravity of mercury.

60 = sec/min.

100 = Conversion to percent.

6.2 Average dry gas meter temperature and average orifice pressure drop. See data sheet (Figure 8–2).

6.3 Dry Gas Volume. Correct the sample volume measured by the dry gas meter to standard conditions (20° C and 760 mm Hg or 68° F and 29.92 in. Hg) by using Equation 8–1.

$$V_{m(std)} = V_m Y \left(\frac{T_{std}}{T_m}\right) \frac{P_{bar} + \left(\frac{\Delta H}{13.6}\right)}{P_{std}}$$

$$= K_1 V_m Y \frac{P_{bar} + (\Delta H/13.6)}{T_m}$$

Equation 8–1

PLANT ____________

LOCATION ____________

OPERATOR ____________

DATE ____________

RUN NO. ____________

SAMPLE BOX NO. ____________

METER BOX NO. ____________

METER $\Delta H_@$ ____________

C FACTOR ____________

PITOT TUBE COEFFICIENT, $C_p$ ____________

SCHEMATIC OF STACK CROSS SECTION

STATIC PRESSURE, mm Hg (in. Hg) ____________

AMBIENT TEMPERATURE ____________

BAROMETRIC PRESSURE ____________

ASSUMED MOISTURE, % ____________

PROBE LENGTH, m (ft) ____________

NOZZLE IDENTIFICATION NO. ____________

AVERAGE CALIBRATED NOZZLE DIAMETER, cm (in.) ____________

PROBE HEATER SETTING ____________

LEAK RATE, $m^3$/min, (cfm) ____________

PROBE LINER MATERIAL ____________

FILTER NO. ____________

| TRAVERSE POINT NUMBER | SAMPLING TIME ($\theta$), min. | VACUUM mm Hg (in. Hg) | STACK TEMPERATURE ($T_S$), °C (°F) | VELOCITY HEAD ($\Delta P_S$), mm $H_2O$ (in. $H_2O$) | PRESSURE DIFFERENTIAL ACROSS ORIFICE METER, mm $H_2O$ (in. $H_2O$) | GAS SAMPLE VOLUME, $m^3$ ($ft^3$) | GAS SAMPLE TEMPERATURE AT DRY GAS METER | | TEMPERATURE OF GAS LEAVING CONDENSER OR LAST IMPINGER, °C (°F) |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | INLET, °C (°F) | OUTLET, °C (°F) | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| TOTAL | | | | | | | Avg | Avg | |
| AVERAGE | | | | | | | Avg | | |

Figure 8-2. Field data.

where:
$K_1$=0.3858 °K/mm Hg for metric units.
=17.64 °R/in. Hg for English units.

NOTE.—If the leak rate observed during any mandatory leak-checks exceeds the specified acceptable rate, the tester shall either correct the value of $V_m$ in Equation 8-1 (as described in Section 6.3 of Method 5), or shall invalidate the test run.

6.4 Volume of Water Vapor and Moisture Content. Calculate the volume of water vapor using Equation 5-2 of Method 5; the weight of water collected in the impingers and silica gel can be directly converted to milliliters (the specific gravity of water is 1 g/ml). Calculate the moisture content of the stack gas, using Equation 5-3 of Method 5. The "Note" in Section 6.5 of Method 5 also applies to this method. Note that if the effluent gas stream can be considered dry, the volume of water vapor and moisture content need not be calculated.

6.5 Sulfuric acid mist (including $SO_3$) concentration.

$$C_{H_2SO_4}=K_2\,\frac{N(V_t-V_{tb})\left(\frac{V_{soln}}{V_a}\right)}{V_{m\,(std)}}$$

Equation 8-2

where:
$K_2$=0.04904 g/milliequivalent for metric units.
=1.081×$10^{-4}$ lb/meq for English units.

6.6 Sulfur dioxide concentration.

$$C_{SO_2}=K_3\,\frac{N(V_t-V_{tb})\left(\frac{V_{soln}}{V_a}\right)}{V_{m\,(std)}}$$

Equation 8-3

where:
$K_3$=0.03203 g/meq for metric units.
=7.061×$10^{-5}$ lb/meq for English units.

6.7 Isokinetic Variation.

6.7.1 Calculation from raw data.

$$I=\frac{100\,T_s[K_4V_{lc}+(V_m/T_m)\,P_{bar}+\Delta H/13.6)]}{60\,\theta V_s P_s A_n}$$

Equation 8-4

where:
$K_4$=0.003464 mm Hg-m³/ml-°K for metric units.
=0.002676 in. Hg-ft³/ml-°R for English units.

6.7.2 Calculation from intermediate values.

$$I=\frac{T_sV_{m\,(std)}\,P_{std}\,100}{T_{std}v_s\theta\,A_nP_s\,60\,(1-B_{ws})}$$

$$=K_5\,\frac{T_sV_{m\,(std)}}{P_sv_sA_n\theta\,(1-B_{ws})}$$

Equation 8-5

where:
$K_5$=4.320 for metric units.
=0.09450 for English units.

6.8 Acceptable Results. If 90 percent ≤I ≤110 percent, the results are acceptable. If the results are low in comparison to the standards and I is beyond the acceptable range, the Administrator may opt to accept the results. Use Citation 4 in the Bibliography of Method 5 to make judgments. Otherwise, reject the results and repeat the test.

7. *Bibliography*

1. Atmospheric Emissions from Sulfuric Acid Manufacturing Processes. U.S. DHEW, PHS, Division of Air Pollution. Public Health Service Publication No. 999-AP-13. Cincinnati, Ohio. 1965.

2. Corbett, P. F. The Determination of $SO_2$ and $SO_3$ in Flue Gases. Journal of the Institute of Fuel. 24:237-243. 1961.

3. Martin, Robert M. Construction Details of Isokinetic Source Sampling Equipment. Environmental Protection Agency. Research Triangle Park, N.C. Air Pollution Control Office Publication No. APTD-0581. April, 1971.

4. Patton, W. F. and J. A. Brink, Jr. New Equipment and Techniques for Sampling Chemical Process Gases. Journal of Air Pollution Control Association. 13:162. 1963.

5. Rom, J. J. Maintenance, Calibration, and Operation of Isokinetic Source-Sampling Equipment. Office of Air Programs, Environmental Protection Agency. Research Triangle Park, N.C. APTD-0576. March, 1972.

6. Hamil, H. F. and D. E. Camann. Collaborative Study of Method for Determination of Sulfur Dioxide Emissions from Stationary Sources (Fossil Fuel-Fired Steam Generators). Environmental Protection Agency. Research Triangle Park, N.C. EPA-650/4-74-024. December, 1973.

7. Annual Book of ASTM Standards. Part 31; Water, Atmospheric Analysis. pp. 40-42. American Society for Testing and Materials. Philadelphia, Pa. 1974.

# METHOD 9

## VISUAL DETERMINATION OF THE OPACITY OF EMISSIONS FROM STATIONARY SOURCES

Many stationary sources discharge visible emissions into the atmosphere; these emissions are usually in the shape of a plume. This method involves the determination of plume opacity by qualified observers. The method includes procedures for the training and certification of observers, and procedures to be used in the field for determination of plume opacity. The appearance of a plume as viewed by an observer depends upon a number of variables, some of which may be controllable and some of which may not be controllable in the field. Variables which can be controlled to an extent to which they no longer exert a significant influence upon plume appearance include: Angle of the observer with respect to the plume; angle of the observer with respect to the sun; point of observation of attached and detached steam plume; and angle of the observer with respect to a plume emitted from a rectangular stack with a large length to width ratio. The method includes specific criteria applicable to these variables.

Other variables which may not be controllable in the field are luminescence and color contrast between the plume and the background against which the plume is viewed. These variables exert an influence upon the appearance of a plume as viewed by an observer, and can affect the ability of the observer to accurately assign opacity values to the observed plume. Studies of the theory of plume opacity and field studies have demonstrated that a plume is most visible and presents the greatest apparent opacity when viewed against a contrasting background. It follows from this, and is confirmed by field trials, that the opacity of a plume, viewed under conditions where a contrasting background is present can be assigned with the greatest degree of accuracy. However, the potential for a positive error is also the greatest when a plume is viewed under such contrasting conditions. Under conditions presenting a less contrasting background, the apparent opacity of a plume is less and approaches zero as the color and luminescence contrast decrease toward zero. As a result, significant negative bias and negative errors can be made when a plume is viewed under less contrasting conditions. A negative bias decreases rather than increases the possibility that a plant operator will be cited for a violation of opacity standards due to observer error

Studies have been undertaken to determine the magnitude of positive errors which can be made by qualified observers while reading plumes under contrasting conditions and using the procedures set forth in this method. The results of these studies (field trials) which involve a total of 769 sets of 25 readings each are as follows:

(1) For black plumes (133 sets at a smoke generator), 100 percent of the sets were read with a positive error[1] of less than 7.5 percent opacity; 99 percent were read with a positive error of less than 5 percent opacity.

(2) For white plumes (170 sets at a smoke generator, 168 sets at a coal-fired power plant, 298 sets at a sulfuric acid plant), 99 percent of the sets were read with a positive error of less than 7.5 percent opacity; 95 percent were read with a positive error of less than 5 percent opacity.

The positive observational error associated with an average of twenty-five readings is therefore established. The accuracy of the method must be taken into account when determining possible violations of applicable opacity standards.

1. *Principle and applicability.*

1.1 Principle. The opacity of emissions from stationary sources is determined visually by a qualified observer.

1.2 Applicability. This method is applicable for the determination of the opacity of emissions from stationary sources pursuant to § 60.11(b) and for qualifying observers for visually determining opacity of emissions.

2. *Procedures.* The observer qualified in accordance with paragraph 3 of this method shall use the following procedures for visually determining the opacity of emissions:

2.1 Position. The qualified observer shall stand at a distance sufficient to provide a clear view of the emissions with the sun oriented in the 140° sector to his back. Consistent with maintaining the above requirement, the observer shall, as much as possible, make his observations from a position such that his line of vision is approximately perpendicular to the plume direction, and when observing opacity of emissions from rectangular outlets (e.g. roof monitors, open baghouses, noncircular stacks), approximately perpendicular to the longer axis of the outlet. The observer's line of sight should not include more than one plume at a time when multiple stacks are involved, and in any case the observer should make his observations with his line of sight perpendicular to the longer axis of such a set of multiple stacks (e.g. stub stacks on baghouses).

2.2 Field records. The observer shall record the name of the plant, emission location, type facility, observer's name and affiliation, and the date on a field data sheet (Figure 9–1). The time, estimated distance to the emission location, approximate wind direction, estimated wind speed, description of the sky condition (presence and color of clouds), and plume background are recorded on a field data sheet at the time opacity readings are initiated and completed.

2.3 Observations. Opacity observations shall be made at the point of greatest opacity in that portion of the plume where condensed water vapor is not present. The observer shall not look continuously at the plume, but instead shall observe the plume momentarily at 15-second intervals.

---

[1] For a set, positive error = average opacity determined by observers' 25 observations − average opacity determined from transmissometer's 25 recordings.

2.3.1 Attached steam plumes. When condensed water vapor is present within the plume as it emerges from the emission outlet, opacity observations shall be made beyond the point in the plume at which condensed water vapor is no longer visible. The observer shall record the approximate distance from the emission outlet to the point in the plume at which the observations are made.

2.3.2 Detached steam plume. When water vapor in the plume condenses and becomes visible at a distinct distance from the emission outlet, the opacity of emissions should be evaluated at the emission outlet prior to the condensation of water vapor and the formation of the steam plume.

2.4 Recording observations. Opacity observations shall be recorded to the nearest 5 percent at 15-second intervals on an observational record sheet. (See Figure 9-2 for an example.) A minimum of 24 observations shall be recorded. Each momentary observation recorded shall be deemed to represent the average opacity of emissions for a 15-second period.

2.5 Data Reduction. Opacity shall be determined as an average of 24 consecutive observations recorded at 15-second intervals. Divide the observations recorded on the record sheet into sets of 24 consecutive observations. A set is composed of any 24 consecutive observations. Sets need not be consecutive in time and in no case shall two sets overlap. For each set of 24 observations, calculate the average by summing the opacity of the 24 observations and dividing this sum by 24. If an applicable standard specifies an averaging time requiring more than 24 observations, calculate the average for all observations made during the specified time period. Record the average opacity on a record sheet. (See Figure 9-1 for an example.)

3. *Qualifications and testing.*

3.1 Certification requirements. To receive certification as a qualified observer, a candidate must be tested and demonstrate the ability to assign opacity readings in 5 percent increments to 25 different black plumes and 25 different white plumes, with an error not to exceed 15 percent opacity on any one reading and an average error not to exceed 7.5 percent opacity in each category. Candidates shall be tested according to the procedures described in paragraph 3.2. Smoke generators used pursuant to paragraph 3.2 shall be equipped with a smoke meter which meets the requirements of paragraph 3.3.

The certification shall be valid for a period of 6 months, at which time the qualification procedure must be repeated by any observer in order to retain certification.

3.2 Certification procedure. The certification test consists of showing the candidate a complete run of 50 plumes—25 black plumes and 25 white plumes—generated by a smoke generator. Plumes within each set of 25 black and 25 white runs shall be presented in random order. The candidate assigns an opacity value to each plume and records his observation on a suitable form. At the completion of each run of 50 readings, the score of the candidate is determined. If a candidate fails to qualify, the complete run of 50 readings must be repeated in any retest. The smoke test may be administered as part of a smoke school or training program, and may be preceded by training or familiarization runs of the smoke generator during which candidates are shown black and white plumes of known opacity.

3.3 Smoke generator specifications. Any smoke generator used for the purposes of paragraph 3.2 shall be equipped with a smoke meter installed to measure opacity across the diameter of the smoke generator stack. The smoke meter output shall display in-stack opacity based upon a pathlength equal to the stack exit diameter, on a full 0 to 100 percent chart recorder scale. The smoke meter optical design and performance shall meet the specifications shown in Table 9-1. The smoke meter shall be calibrated as prescribed in paragraph 3.3.1 prior to the conduct of each smoke reading test. At the completion of each test, the zero and span drift shall be checked and if the drift exceeds ±1 percent opacity, the condition shall be corrected prior to conducting any subsequent test runs. The smoke meter shall be demonstrated, at the time of installation, to meet the specifications listed in Table 9-1. This demonstration shall be repeated following any subsequent repair or replacement of the photocell or associated electronic circuitry including the chart recorder or output meter, or every 6 months, whichever occurs first.

TABLE 9-1—SMOKE METER DESIGN AND PERFORMANCE SPECIFICATIONS

| Parameter: | *Specification* |
|---|---|
| a. Light source | Incandescent lamp operated at nominal rated voltage. |
| b. Spectral response of photocell. | Photopic (daylight spectral response of the human eye—reference 4.3). |
| c. Angle of view | 15° maximum total angle. |
| d. Angle of projection. | 15° maximum total angle. |
| e. Calibration error. | ±3% opacity, maximum. |
| f. Zero and span drift. | ±1% opacity, 30 minutes. |
| g. Response time | ≤5 seconds. |

3.3.1 Calibration. The smoke meter is calibrated after allowing a minimum of 30 minutes warmup by alternately producing simulated opacity of 0 percent and 100 percent. When stable response at 0 percent or 100 percent is noted, the smoke meter is adjusted to produce an output of 0 percent or 100 percent, as appropriate. This calibration shall be repeated until stable 0 percent and 100 percent readings are produced without adjustment. Simulated 0 percent and 100 percent opacity values may be produced by alternately switching the power to the light source on and off while the smoke generator is not producing smoke.

3.3.2 Smoke meter evaluation. The smoke meter design and performance are to be evaluated as follows:

3.3.2.1 Light source. Verify from manufacturer's data and from voltage measurements made at the lamp, as installed, that the lamp is operated within ±5 percent of the nominal rated voltage.

3.3.2.2 Spectral response of photocell. Verify from manufacturer's data that the photocell has a photopic response; i.e., the spectral sensitivity of the cell shall closely approximate the standard spectral-luminosity curve for photopic vision which is referenced in (b) of Table 9-1.

3.3.2.3 Angle of view. Check construction geometry to ensure that the total angle of view of the smoke plume, as seen by the photocell, does not exceed 15°. The total angle of view may be calculated from: $\theta=2 \tan^{-1} d/2L$, where $\theta$=total angle of view; d=the sum of the photocell diameter+the diameter of the limiting aperture; and L=the distance from the photocell to the limiting aperture. The limiting aperture is the point in the path between the photocell and the smoke plume where the angle of view is most restricted. In smoke generator smoke meters this is normally an orifice plate.

3.3.2.4 Angle of projection. Check construction geometry to ensure that the total angle of projection of the lamp on the smoke plume does not exceed 15°. The total angle of projection may be calculated from: $\theta=2 \tan^{-1} d/2L$, where $\theta$= total angle of projection; d= the sum of the length of the lamp filament + the diameter of the limiting aperture; and L= the distance from the lamp to the limiting aperture.

3.3.2.5 Calibration error. Using neutral-density filters of known opacity, check the error between the actual response and the theoretical linear response of the smoke meter. This check is accomplished by first calibrating the smoke meter according to 3.3.1 and then inserting a series of three neutral-density filters of nominal opacity of 20, 50, and 75 percent in the smoke meter pathlength. Filters calibarted within ±2 percent shall be used. Care should be taken when inserting the filters to prevent stray light from affecting the meter. Make a total of five nonconsecutive readings for each filter. The maximum error on any one reading shall be 3 percent opacity.

3.3.2.6 Zero and span drift. Determine the zero and span drift by calibrating and operating the smoke generator in a normal manner over a 1-hour period. The drift is measured by checking the zero and span at the end of this period.

3.3.2.7 Response time. Determine the response time by producng the series of five simulated 0 percent and 100 percent opacity values and observing the time required to reach stable response. Opacity values of 0 percent and 100 percent may be simulated by alternately switching the power to the light source off and on while the smoke generator is not operating.

4. *References.*

4.1 Air Pollution Control District Rules and Regulations, Los Angeles County Air Pollution Control District, Regulation IV, Prohibitions, Rule 50.

4.2 Weisburd, Melvin I., Field Operations and Enforcement Manual for Air, U.S. Environmental Protection Agency, Research Triangle Park, N.C., APTD-1100, August 1972. pp. 4.1-4.36.

4.3 Condon, E. U., and Odishaw, H., Handbook of Physics, McGraw-Hill Co., N.Y., N.Y., 1958, Table 3.1, p. 6-52.

FIGURE 9-1
RECORD OF VISUAL DETERMINATION OF OPACITY

PAGE___of___

COMPANY________________
LOCATION________________
TEST NUMBER________________
DATE________________
TYPE FACILITY________________
CONTROL DEVICE________________

HOURS OF OBSERVATION________________
OBSERVER________________
OBSERVER CERTIFICATION DATE________________
OBSERVER AFFILIATION________________
POINT OF EMISSIONS________________
HEIGHT OF DISCHARGE POINT________________

---

| CLOCK TIME | Initial | | | Final |
|---|---|---|---|---|
| OBSERVER LOCATION<br>Distance to Discharge | | | | |
| Direction from Discharge | | | | |
| Height of Observation Point | | | | |
| BACKGROUND DESCRIPTION | | | | |
| WEATHER CONDITIONS<br>Wind Direction | | | | |
| Wind Speed | | | | |
| Ambient Temperature | | | | |
| SKY CONDITIONS (clear, overcast, % clouds, etc.) | | | | |
| PLUME DESCRIPTION<br>Color | | | | |
| Distance Visible | | | | |
| OTHER INFORMATION | | | | |

SUMMARY OF AVERAGE OPACITY

| Set Number | Time | Opacity | |
|---|---|---|---|
| | Start--End | Sum | Average |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |

Readings ranged from ____ to ____ % opacity

The source was/was not in compliance with ____ at the time evaluation was made.

Reproduced from best available copy.

FIGURE 9-2 OBSERVATION RECORD PAGE ___ OF ___

COMPANY ____________________
LOCATION ____________________
TEST NUMBER ____________________
DATE ____________________

OBSERVER ____________________
TYPE FACILITY ____________________
POINT OF EMISSIONS ____________________

| | | Seconds | | | | STEAM PLUME (check if applicable) | | COMMENTS |
|---|---|---|---|---|---|---|---|---|
| Hr. | Min. | 0 | 15 | 30 | 45 | Attached | Detached | |
| | 0 | | | | | | | |
| | 1 | | | | | | | |
| | 2 | | | | | | | |
| | 3 | | | | | | | |
| | 4 | | | | | | | |
| | 5 | | | | | | | |
| | 6 | | | | | | | |
| | 7 | | | | | | | |
| | 8 | | | | | | | |
| | 9 | | | | | | | |
| | 10 | | | | | | | |
| | 11 | | | | | | | |
| | 12 | | | | | | | |
| | 13 | | | | | | | |
| | 14 | | | | | | | |
| | 15 | | | | | | | |
| | 16 | | | | | | | |
| | 17 | | | | | | | |
| | 18 | | | | | | | |
| | 19 | | | | | | | |
| | 20 | | | | | | | |
| | 21 | | | | | | | |
| | 22 | | | | | | | |
| | 23 | | | | | | | |
| | 24 | | | | | | | |
| | 25 | | | | | | | |
| | 26 | | | | | | | |
| | 27 | | | | | | | |
| | 28 | | | | | | | |
| | 29 | | | | | | | |

# METHOD 10

# DETERMINATION OF CARBON MONOXIDE EMISSIONS FROM STATIONARY SOURCES

1. *Principle and Applicability.*

1.1 *Principle.* An integrated or continuous gas sample is extracted from a sampling point and analyzed for carbon monoxide (CO) content using a Luft-type nondispersive infrared analyzer (NDIR) or equivalent.

1.2 *Applicability.* This method is applicable for the determination of carbon monoxide emissions from stationary sources only when specified by the test procedures for determining compliance with new source performance standards. The test procedure will indicate whether a continuous or an integrated sample is to be used.

2. *Range and sensitivity.*

2.1 *Range.* 0 to 1,000 ppm.

2.2 *Sensitivity.* Minimum detectable concentration is 20 ppm for a 0 to 1,000 ppm span.

3. *Interferences.* Any substance having a strong absorption of infrared energy will interfere to some extent. For example, discrimination ratios for water ($H_2O$) and carbon dioxide ($CO_2$) are 3.5 percent $H_2O$ per 7 ppm CO and 10 percent $CO_2$ per 10 ppm CO, respectively, for devices measuring in the 1,500 to 3,000 ppm range. For devices measuring in the 0 to 100 ppm range, interference ratios can be as high as 3.5 percent $H_2O$ per 25 ppm CO and 10 percent $CO_2$ per 50 ppm CO. The use of silica gel and ascarite traps will alleviate the major interference problems. The measured gas volume must be corrected if these traps are used.

4. *Precision and accuracy.*

4.1 *Precision.* The precision of most NDIR analyzers is approximately ±2 percent of span.

4.2 *Accuracy.* The accuracy of most NDIR analyzers is approximately ±5 percent of span after calibration.

5. *Apparatus.*

5.1 *Continuous sample* (Figure 10–1).

5.1.1 *Probe.* Stainless steel or sheathed Pyrex[1] glass, equipped with a filter to remove particulate matter.

5.1.2 *Air-cooled condenser or equivalent.* To remove any excess moisture.

5.2 *Integrated sample* (Figure 10–2).

5.2.1 *Probe.* Stainless steel or sheathed Pyrex glass, equipped with a filter to remove particulate matter.

5.2.2 *Air-cooled condenser or equivalent.* To remove any excess moisture.

5.2.3 *Valve.* Needle valve, or equivalent, to adjust flow rate.

5.2.4 *Pump.* Leak-free diaphragm type, or equivalent, to transport gas.

5.2.5 *Rate meter.* Rotameter, or equivalent, to measure a flow range from 0 to 1.0 liter per min. (0.035 cfm).

5.2.6 *Flexible bag.* Tedlar, or equivalent, with a capacity of 60 to 90 liters (2 to 3 ft$^3$). Leak-test the bag in the laboratory before using by evacuating bag with a pump followed by a dry gas meter. When evacuation is complete, there should be no flow through the meter.

5.2.7 *Pitot tube.* Type S, or equivalent, attached to the probe so that the sampling rate can be regulated proportional to the stack gas velocity when velocity is varying with the time or a sample traverse is conducted.

5.3 *Analysis* (Figure 10–3).

5.3.1 *Carbon monoxide analyzer.* Nondispersive infrared spectrometer, or equivalent. This instrument should be demonstrated, preferably by the manufacturer, to meet or exceed manufacturer's specifications and those described in this method.

5.3.2 *Drying tube.* To contain approximately 200 g of silica gel.

5.3.3 *Calibration gas.* Refer to paragraph 6.1.

5.3.4 *Filter.* As recommended by NDIR manufacturer.

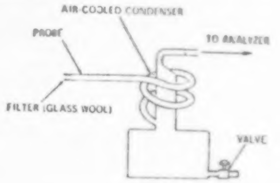


Figure 10-1. Continuous sampling train.

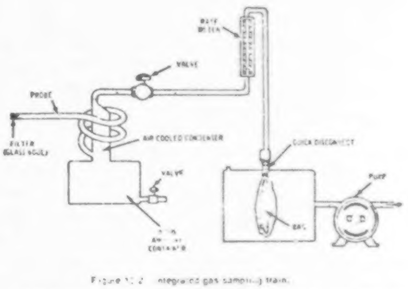


Figure 10-2. Integrated gas sampling train.

5.3.5 *$CO_2$ removal tube.* To contain approximately 500 g of ascarite.

5.3.6 *Ice water bath.* For ascarite and silica gel tubes.

5.3.7 *Valve.* Needle valve, or equivalent, to adjust flow rate

5.3.8 *Rate meter.* Rotameter or equivalent to measure gas flow rate of 0 to 1.0 liter per min. (0.035 cfm) through NDIR.

5.3.9 *Recorder (optional).* To provide permanent record of NDIR readings.

6. *Reagents.*

---

[1] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

FIGURE 9-2 OBSERVATION RECORD PAGE ___ OF __
(Continued)

COMPANY ____________________
LOCATION ____________________
TEST NUMBER ____________________
DATE ____________________

OBSERVER ____________________
TYPE FACILITY ____________________
POINT OF EMISSIONS ____________________

| | | Seconds | | | | STEAM PLUME (check if applicable) | | |
|---|---|---|---|---|---|---|---|---|
| Hr. | Min. | 0 | 15 | 30 | 45 | Attached | Detached | COMMENTS |
| | 30 | | | | | | | |
| | 31 | | | | | | | |
| | 32 | | | | | | | |
| | 33 | | | | | | | |
| | 34 | | | | | | | |
| | 35 | | | | | | | |
| | 36 | | | | | | | |
| | 37 | | | | | | | |
| | 38 | | | | | | | |
| | 39 | | | | | | | |
| | 40 | | | | | | | |
| | 41 | | | | | | | |
| | 42 | | | | | | | |
| | 43 | | | | | | | |
| | 44 | | | | | | | |
| | 45 | | | | | | | |
| | 46 | | | | | | | |
| | 47 | | | | | | | |
| | 48 | | | | | | | |
| | 49 | | | | | | | |
| | 50 | | | | | | | |
| | 51 | | | | | | | |
| | 52 | | | | | | | |
| | 53 | | | | | | | |
| | 54 | | | | | | | |
| | 55 | | | | | | | |
| | 56 | | | | | | | |
| | 57 | | | | | | | |
| | 58 | | | | | | | |
| | 59 | | | | | | | |

6.1 *Calibration gases*. Known concentration of CO in nitrogen ($N_2$) for instrument span, prepurified grade of $N_2$ for zero, and two additional concentrations corresponding approximately to 60 percent and 30 percent span. The span concentration shall not exceed 1.5 times the applicable source performance standard. The calibration gases shall be certified by the manufacturer to be within ±2 percent of the specified concentration.

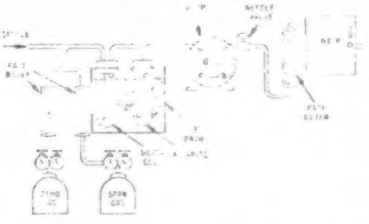


Figure 10-3. Analytical equipment.

6.2 *Silica gel*. Indicating type, 6 to 16 mesh, dried at 175° C (347° F) for 2 hours.

6.3 *Ascarite*. Commercially available.

7. *Procedure*.

7.1 *Sampling*.

7.1.1 *Continuous sampling*. Set up the equipment as shown in Figure 10-1 making sure all connections are leak free. Place the probe in the stack at a sampling point and purge the sampling line. Connect the analyzer and begin drawing sample into the analyzer. Allow 5 minutes for the system to stabilize, then record the analyzer reading as required by the test procedure. (See § 7.2 and 8). $CO_2$ content of the gas may be determined by using the Method 3 integrated sample procedure (36 FR 24886), or by weighing the ascarite $CO_2$ removal tube and computing $CO_2$ concentration from the gas volume sampled and the weight gain of the tube.

7.1.2 *Integrated sampling*. Evacuate the flexible bag. Set up the equipment as shown in Figure 10-2 with the bag disconnected. Place the probe in the stack and purge the sampling line. Connect the bag, making sure that all connections are leak free. Sample at a rate proportional to the stack velocity. $CO_2$ content of the gas may be determined by using the Method 3 integrated sample procedures (36 FR 24886), or by weighing the ascarite $CO_2$ removal tube and computing $CO_2$ concentration from the gas volume sampled and the weight gain of the tube.

7.2 *CO Analysis*. Assemble the apparatus as shown in Figure 10-3, calibrate the instrument, and perform other required operations as described in paragraph 8. Purge analyzer with $N_2$ prior to introduction of each sample. Direct the sample stream through the instrument for the test period, recording the readings. Check the zero and span again after the test to assure that any drift or malfunction is detected. Record the sample data on Table 10-1.

8. *Calibration*. Assemble the apparatus according to Figure 10-3. Generally an instrument requires a warm-up period before stability is obtained. Follow the manufacturer's instructions for specific procedure. Allow a minimum time of one hour for warm-up. During this time check the sample conditioning apparatus, i.e., filter, condenser, drying tube, and $CO_2$ removal tube, to ensure that each component is in good operating condition. Zero and calibrate the instrument according to the manufacturer's procedures using, respectively, nitrogen and the calibration gases.

9. *Calculation—Concentration of carbon monoxide*. Calculate the concentration of carbon monoxide in the stack using equation 10-1.

$$C_{CO_{stack}} = C_{CO_{NDIR}}(1 - F_{CO_2}) \qquad \text{equation 10-1}$$

where:

$C_{CO_{stack}}$ = concentration of CO in stack, ppm by volume (dry basis).

$C_{CO_{NDIR}}$ = concentration of CO measured by NDIR analyzer, ppm by volume (dry basis).

$F_{CO_2}$ = volume fraction of $CO_2$ in sample, i.e., percent $CO_2$ from Orsat analysis divided by 100.

10 *Bibliography*

10.1 McElroy, Frank. The Intertech NDIR-CO Analyzer. Presented at 11th Methods Conference on Air Pollution, University of California, Berkeley, Calif., April 1, 1970.

10.2 Jacobs, M. B., et al., Continuous Determination of Carbon Monoxide and Hydrocarbons in Air by a Modified Infrared Analyzer, J. Air Pollution Control Association, 9(2):110–114, August 1959.

10.3 MSA LIRA Infrared Gas and Liquid Analyzer Instruction Book, Mine Safety Appliances Co., Technical Products Division, Pittsburgh, Pa.

10.4 Models 215A, 315A, and 415A Infrared Analyzers, Beckman Instruments, Inc., Beckman Instructions 1635-B, Fullerton, Calif., October 1967.

10.5 Continuous CO Monitoring System, Model A5611, Intertech Corp., Princeton, N.J.

10.6 UNOR Infrared Gas Analyzers, Bendix Corp., Ronceverte, West Virginia.

TABLE 10-1.—*Field data*

**Location** ---------------------------------------------------- Comments:
**Test** ----------------------------------------------------
**Date** ----------------------------------------------------
**Operator** ----------------------------------------------------

| *Clock time* | *Rotameter setting, liters per minute (cubic feet per minute)* |
|---|---|
| | |

## ADDENDA

### *A. Performance Specifications for NDIR Carbon Monoxide Analyzers.*

| | |
|---|---|
| Range (minimum) | 0–1000ppm. |
| Output (minimum) | 0–10mV. |
| Minimum detectable sensitivity | 20 ppm. |
| Rise time, 90 percent (maximum) | 30 seconds. |
| Fall time, 90 percent (maximum) | 30 seconds. |
| Zero drift (maximum) | 10% in 8 hours. |
| Span drift (maximum) | 10% in 8 hours. |
| Precision (minimum) | ± 2% of full scale. |
| Noise (maximum) | ± 1% of full scale. |
| Linearity (maximum deviation) | 2% of full scale. |
| Interference rejection ratio | $CO_2$—1000 to 1, $H_2O$—500 to 1. |

### B. *Definitions of Performance Specifications.*

*Range*—The minimum and maximum measurement limits.

*Output*—Electrical signal which is proportional to the measurement; intended for connection to readout or data processing devices. Usually expressed as millivolts or milliamps full scale at a given impedance.

*Full scale*—The maximum measuring limit for a given range.

*Minimum detectable sensitivity*—The smallest amount of input concentration that can be detected as the concentration approaches zero.

*Accuracy*—The degree of agreement between a measured value and the true value; usually expressed as ± percent of full scale.

*Time to 90 percent response*—The time interval from a step change in the input concentration at the instrument inlet to a reading of 90 percent of the ultimate recorded concentration.

*Rise Time (90 percent)*—The interval between initial response time and time to 90 percent response after a step increase in the inlet concentration.

*Fall Time (90 percent)*—The interval between initial response time and time to 90 percent response after a step decrease in the inlet concentration.

*Zero Drift*—The change in instrument output over a stated time period, usually 24 hours, of unadjusted continuous operation when the input concentration is zero; usually expressed as percent full scale.

*Span Drift*—The change in instrument output over a stated time period, usually 24 hours, of unadjusted continuous operation when the input concentration is a stated upscale value; usually expressed as percent full scale.

*Precision*—The degree of agreement between repeated measurements of the same concentration, expressed as the average deviation of the single results from the mean.

*Noise*—Spontaneous deviations from a mean output not caused by input concentration changes.

*Linearity*—The maximum deviation between an actual instrument reading and the reading predicted by a straight line drawn between upper and lower calibration points.

# METHOD 11

# DETERMINATION OF HYDROGEN SULFIDE EMISSIONS FROM STATIONARY SOURCES

1. *Principle and applicability.*

1.1 *Principle.* Hydrogen sulfide ($H_2S$) is collected from the source in a series of midget impingers and reacted with alkaline cadmium hydroxide [$Cd(OH)_2$] to form cadmium sulfide (CdS). The precipitated CdS is then dissolved in hydrochloric acid and absorbed in a known volume of iodine solution. The iodine consumed is a measure of the $H_2S$ content of the gas. An impinger containing hydrogen peroxide is included to remove $SO_2$ as an interfering species.

1.2 *Applicability.* This method is applicable for the determination of hydrogen sulfide emissions from stationary sources only when specified by the test procedures for determining compliance with the new source performance standards.

2. *Apparatus.*

2.1 *Sampling train.*

2.1.1 *Sampling line*—6- to 7-mm (¼-inch) Teflon[1] tubing to connect sampling train to sampling valve, with provisions for heating to prevent condensation. A pressure reducing valve prior to the Teflon sampling line may be required depending on sampling stream pressure.

2.1.2 *Impingers*—Five midget impingers, each with 30-ml capacity, or equivalent.

2.1.3 *Ice bath container*—To maintain absorbing solution at a constant temperature.

2.1.4 *Silica gel drying tube*—To protect pump and dry gas meter.

2.1.5 *Needle valve, or equivalent*—Stainless steel or other corrosion resistant material, to adjust gas flow rate.

2.1.6 *Pump*—Leak free, diaphragm type, or equivalent, to transport gas. (Not required if sampling stream under positive pressure.)

2.1.7 *Dry gas meter*—Sufficiently accurate to measure sample volume to within 1 percent.

2.1.8 *Rate meter*—Rotameter, or equivalent, to measure a flow rate of 0 to 3 liters per minute (0.1 ft$^3$/min).

2.1.9 *Graduated cylinder*—25 ml.

2.1.10 *Barometer*—To measure atmospheric pressure within ±2.5 mm (0.1 in.) Hg.

2.2 *Sample Recovery.*

2.2.1 *Sample container*—500-ml glass-stoppered iodine flask.

2.2.2 *Pipette*—50-ml volumetric type.

2.2.3 *Beakers*—250 ml.

2.2.4 *Wash bottle*—Glass.

2.3 *Analysis.*

2.3.1 *Flask*—500-ml glass-stoppered iodine flask.

2.3.2 *Burette*—One 50 ml.

2.3.2 *Flask*—125-ml conical.

3. *Reagents.*

3.1 *Sampling.*

3.1.1 *Absorbing solution*—Cadmium hydroxide ($Cd(OH)_2$)—Mix 4.3 g cadmium sulfate hydrate ($3\ CdSO_4 \cdot 8H_2O$) and 0.3 g of sodium hydroxide (NaOH) in 1 liter of distilled water ($H_2O$). Mix well.

*Note:* The cadmium hydroxide formed in this mixture will precipitate as a white suspension. Therefore, this solution must be thoroughly mixed before using to ensure an even distribution of the cadmium hydroxide.

3.1.2 *Hydrogen peroxide, 3 percent*—Dilute 30 percent hydrogen peroxide to 3 percent as needed. Prepare fresh daily;

3.2 *Sample recovery.*

3.2.1 *Hydrochloric acid solution (HCl), 10 percent by weight*—mix 230 ml of concentrated HCl (specific gravity 1.19) and 770 ml of distilled $H_2O$.

3.2.2 *Iodine solution, 0.1 N*—Dissolve 24 g potassium iodide (KI) in 30 ml of distilled $H_2O$ in a 1-liter graduated cylinder. Weigh 12.7 g of resublimed iodine ($I_2$) into a weighing bottle and add to the potassium iodide solution. Shake the mixture until the iodine is completely dissolved. Slowly dilute the solution to 1 liter with distilled $H_2O$, with swirling. Filter the solution, if cloudy, and store in a brown glass-stoppered bottle.

3.2.3 *Standard iodine solution, 0.01 N*—Dilute 100 ml of the 0.1 *N* iodine solution in a volumetric flask to 1 liter with distilled water.

Standardize daily as follows: Pipette 25 ml of the 0.01 *N* iodine solution into a 125-ml conical flask. Titrate with standard 0.01 *N* thiosulfate solution (see paragraph 3.3.2) until the solution is a light yellow. Add a few drops of the starch solution and continue titrating until the blue color just disappears. From the results of this titration, calculate the exact normality of the iodine solution (see paragraph 5.1).

3.2.4 *Distilled, deionized water.*

3.3 *Analysis.*

3.3.1 *Sodium thiosulfate solution, standard 0.1 N*—For each liter of solution, dissolve 24.8 g of sodium thiosulfate ($NA_2S_2O_3 \cdot 5H_2O$) in distilled water and add 0.01 g of anhydrous sodium carbonate ($Na_2CO_3$) and 0.4 ml of chloroform ($CHCl_3$) to stabilize. Mix thoroughly by shaking or by aerating with nitrogen for approximately 15 minutes, and store in a glass-stoppered glass bottle.

Standardize frequently as follows: Weigh into a 500-ml volumetric flask about 2 g of potassium dichromate ($K_2Cr_2O_7$) weighed to the nearest milligram and dilute to the 500-ml mark with distilled $H_2O$. Use dichromate which has been crystallized from distilled water and oven-dried at 182°C to 199°C (360°F to 390°F). Dissolve approximately 3 g of potassium iodide (KI) in 50 ml of distilled water in a glass-stoppered, 500-ml conical flask, then add 5 ml of 20-percent hydrochloric acid solution. Pipette 50 ml of the dichromate solution into this mixture. Gently swirl the solution once and allow it to stand in the dark for 5 minutes. Dilute the solution with 100 to 200 ml of distilled water, washing down the sides of the flask with part of the water. Swirl the solution slowly and titrate with the thiosulfate solution until the solution is light yellow. Add 4 ml of starch solution and continue with a slow titration with the thiosulfate until the bright blue color has disappeared and only the pale green color of the chromic ion remains. From this titration, calculate the exact normality of the sodium thiosulfate solution (see paragraph 5.2).

---

[1] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

3.3.2 *Sodium thiosulfate solution, standard 0.01 N*—Pipette 100 ml of the standard 0.1 *N* thiosulfate solution into a volumetric flask and dilute to one liter with distilled water.

3.3.3 *Starch indicator solution*—Suspend 10 g of soluble starch in 100 ml of distilled water and add 15 g of potassium hydroxide pellets. Stir until dissolved, dilute with 900 ml of distilled water, and let stand 1 hour. Neutralize the alkali with concentrated hydrochloric acid, using an indicator paper similar to Alkacid test ribbon, then add 2 ml of glacial acetic acid as a preservative.

Test for decomposition by titrating 4 ml of starch solution in 200 ml of distilled water with 0.01 *N* iodine solution. If more than 4 drops of the 0.01 *N* iodine solution are required to obtain the blue color, make up a fresh starch solution.

4. *Procedure.*

4.1 *Sampling.*

4.1.1 Assemble the sampling train as shown in Figure 11-1, connecting the five midget impingers in series. Place 15 ml of 3 percent hydrogen peroxide in the first impinger. Place 15 ml of the absorbing solution in each of the next three impingers, leaving the fifth dry. Place crushed ice around the impingers. Add more ice during the run to keep the temperature of the gases leaving the last impinger at about 20°C (70°F), or less.

4.1.2 Purge the connecting line between the sampling valve and the first impinger. Connect the sample line to the train. Record the initial reading on the dry gas meter as shown in Table 11-1.

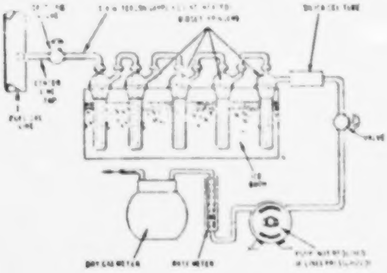


Figure 11-1. $H_2S$ sampling train.

TABLE 11-1.—*Field data*

Location ____________ Comments:
Test ________________
Date ________________
Operator ____________
Barometric pressure __

| Clock time | Gas volume through meter ($V_m$), liters (cubic feet) | Rotameter setting, Lpm (cubic feet per minute) | Meter temperature, °C (°F) |
|---|---|---|---|
| | | | |

4.1.3 Open the flow control valve and adjust the sampling rate to 1.13 liters per minute (0.04 cfm). Read the meter temperature and record on Table 11-1.

4.1.4 Continue sampling a minimum of 10 minutes. If the yellow color of cadmium sulfide is visible in the third impinger, analysis should confirm that the applicable standard has been exceeded. At the end of the sample time, close the flow control valve and read the final meter volume and temperature.

4.1.5 Disconnect the impinger train from the sampling line. Purge the train with clean ambient air for 15 minutes to ensure that all $H_2S$ is removed from the hydrogen peroxide. Cap the open ends and move to the sample clean-up area.

4.2 *Sample recovery.*

4.2.1 Pipette 50 ml of 0.01 *N* iodine solution into a 250-ml beaker. Add 50 ml of 10 percent HCl to the solution. Mix well.

4.2.2 Discard the contents of the hydrogen peroxide impinger. Carefully transfer the contents of the remaining four impingers to a 500-ml iodine flask.

4.2.3 Rinse the four absorbing impingers and connecting glassware with three portions of the acidified iodine solution. Use the entire 100 ml of acidified iodine for this purpose. Immediately after pouring the acidified iodine into an impinger, stopper it and shake for a few moments before transferring the rinse to the iodine flask. Do not transfer any rinse portion from one impinger to another; transfer it directly to the iodine flask. Once acidified iodine solution has been poured into any glassware containing cadmium sulfide sample, the container must be tightly stoppered at all times except when adding more solution, and this must be done as quickly and carefully as possible. After adding any acidified iodine solution to the iodine flask, allow a few minutes for absorption of the $H_2S$ into the iodine before adding any further rinses.

4.2.4 Follow this rinse with two more rinses using distilled water. Add the distilled water rinses to the iodine flask. Stopper the flask and shake well. Allow about 30 minutes for absorption of the $H_2S$ into the iodine, then complete the analysis titration.

*Caution:* Keep the iodine flask stoppered except when adding sample or titrant.

4.2.5 Prepare a blank in an iodine flask using 45 ml of the absorbing solution, 50 ml of 0.01 *N* iodine solution, and 50 ml of 10 percent HCl. Stopper the flask, shake well and analyze with the samples.

4.3 *Analysis.*

*Note:* This analysis titration should be conducted at the sampling location in order to prevent loss of iodine from the sample. Titration should never be made in direct sunlight.

4.3.1 Titrate the solution in the flask with 0.01 *N* sodium thiosulfate solution until the solution is light yellow. Add 4 ml of the starch indicator solution and continue titrating until the blue color just disappears.

4.3.2 Titrate the blanks in the same manner as the samples.

5. *Calculations.*

5.1 *Normality of the standard iodine solution.*

$$N_I=\frac{N_T V_T}{V_I} \qquad \text{equation 11-1}$$

where:

$N_I$ = normality of iodine, g-eq/liter.
$V_I$ = volume of iodine used, ml.
$N_T$ = normality of sodium thiosulfate, g-eq/liter.
$V_T$ = volume of sodium thiosulfate used, ml.

5.2 *Normality of the standard thiosulfate solution.*

$$N_T = 2.04\,\frac{W}{V_T} \qquad \text{equation 11-2}$$

where:

$W$ = weight of $K_2Cr_2O_7$ used, g.
$V_T$ = volume of $Na_2S_2O_3$ used, ml.
$N_T$ = normality of standard thiosulfate solution, g-eq/liter.
2.04 = conversion factor

$$= \frac{(6\ \text{eq}\ I_2/\text{mole}\ K_2Cr_2O_7)\ (1{,}000\ \text{ml/l})}{(294.2\ \text{g}\ K_2Cr_2O_7/\text{mole})\ (10\ \text{aliquot factor})}.$$

5.3 *Dry gas volume.* Correct the sample volume measured by the dry gas meter to standard conditions [21°C(70°F)] and 760 mm (29.92 inches) Hg] by using equation 11-3.

$$V_{m_{std}} = V_m \left(\frac{T_{std}}{T_m}\right)\left(\frac{P_{bar}}{P_{std}}\right) \qquad \text{equation 11-3}$$

where:

$V_{m_{std}}$ = volume at standard conditions of gas sample through the dry gas meter, standard liters (scf).
$V_m$ = volume of gas sample through the dry gas meter (meter conditions), liters (cu. ft.).
$T_{std}$ = absolute temperature at standard conditions, 294°K (530°R).
$T_m$ = average dry gas meter temperature, °K (°R).
$P_{bar}$ = barometric pressure at the orifice meter, mm Hg (in. Hg).
$P_{std}$ = absolute pressure at standard conditions, 760 mm Hg (29.92 in. Hg).

5.4 *Concentration of $H_2S$.*—Calculate the concentration of $H_2S$ in the gas stream at standard conditions using equation 11-4:

$$C_{H_2S} = \frac{K[(V_I N_I - V_T N_T)_{sample} - (V_I N_I - V_T N_T)_{blank}]}{V_{m_{std}}}$$

where (metric units):

$C_{H_2S}$ = concentration of $H_2S$ at standard conditions, mg/dscm
$K$ = conversion factor = $17.0 \times 10^3$

$$= \frac{(34.07\ \text{g/mole}\ H_2S)(1{,}000\ \text{l}/m^3)(1{,}000\ \text{mg/g})}{(1{,}000\ \text{ml/l})(2H_2S\ \text{eq/mole})}$$

$V_I$ = volume of standard iodine solution, ml.
$N_I$ = normality of standard iodine solution, g-eq/liter.
$V_T$ = volume of standard sodium thiosulfate solution, ml.
$N_T$ = normality of standard sodium thiosulfate solution, g-eq/liter.
$V_{m_{std}}$ = dry gas volume at standard conditions, liters.

where (English units):

$$K = 0.263 = \frac{17.0(15.43\ \text{gr/g})}{(1{,}000\ \text{l/m}^3)}$$

$V_{m_{std}}$ = scf.
$C_{H_2S}$ = gr/dscf.

6. *References.*

6.1 Determination of Hydrogen Sulfide, Ammoniacal Cadmium Chloride Method, API Method 772-54. In: Manual on Disposal of Refinery Wastes, Vol. V: Sampling and Analysis of Waste Gases and Particulate Matter, American Petroleum Institute, Washington, D.C., 1954.

6.2 Tentative Method for Determination of Hydrogen Sulfide and Mercaptan Sulfur in Natural Gas, Natural Gas Processors Association, Tulsa, Oklahoma, NGPA Publication No. 2265-65, 1965.

# METHOD 13A

# DETERMINATION OF TOTAL FLUORIDE EMISSIONS FROM STATIONARY SOURCES - SPADNS ZIRCONIUM LAKE METHOD

1. *Principle and Applicability.*

1.1 *Principle.* Gaseous and particulate fluorides are withdrawn isokinetically from the source using a sampling train. The fluorides are collected in the impinger water and on the filter of the sampling train. The weight of total fluorides in the train is determined by the SPADNS Zirconium Lake colorimetric method.

1.2 *Applicability.* This method is applicable for the determination of fluoride emissions from stationary sources only when specified by the test procedures for determining compliance with new source performance standards. Fluorocarbons, such as Freons, are not quantitatively collected or measured by this procedure.

2. *Range and Sensitivity.*

The SPADNS Zirconium Lake analytical method covers the range from 0-1.4 μg/ml fluoride. Sensitivity has not been determined.

3. *Interferences.*

During the laboratory analysis, aluminum in excess of 300 mg/liter and silicon dioxide in excess of 300 μg/liter will prevent complete recovery of fluoride. Chloride will distill over and interfere with the SPADNS Zirconium Lake color reaction. If chloride ion is present, use of Specific Ion Electrode (Method 13B) is recommended; otherwise a chloride determination is required and 5 mg of silver sulfate (see section 7.3.6) must be added for each mg of chloride to prevent chloride interference. If sulfuric acid is carried over in the distillation, it will cause a positive interference. To avoid sulfuric acid carryover, it is important to stop distillation at 175°C.

4. *Precision, Accuracy and Stability.*

4.1 *Analysis.* A relative standard deviation of 3 percent was obtained from twenty replicate intralaboratory determinations on stack emission samples with a concentration range of 39 to 360 mg/l. A phosphate rock standard which was analyzed by this procedure contained a certified value of 3.84 percent. The average of five determinations was 3.88 percent fluoride.

4.2 *Stability.* The color obtained when the sample and colorimetric reagent are mixed is stable for approximately two hours. After formation of the color, the absorbances of the sample and standard solutions should be measured at the same temperature. A 3°C temperature difference between sample and standard solutions will produce an error of approximately 0.005 mg F/liter.

5. *Apparatus.*

5.1 *Sample train.* See Figure 13A-1; it is similar to the Method 5 train except for the interchangeability of the position of the filter. Commercial models of this train are available. However, if one desires to build his own, complete construction details are described in APTD-0581; for changes from the APTD-0581 document and for allowable modifications to Figure 13A-1, see the following subsections.

The operating and maintenance procedures for the sampling train are described in APTD-0576. Since correct usage is important in obtaining valid results, all users should read the APTD-0576 document and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein.

5.1.1 Probe nozzle—Stainless steel (316) with sharp, tapered leading edge. The angle of taper shall be ≤30° and the taper shall be on the outside to preserve a constant internal diameter. The probe nozzle shall be of the button-hook or elbow design, unless otherwise specified by the Administrator. The wall thickness of the nozzle shall be less than or equal to that of 20 gauge tubing, i.e., 0.165 cm (0.065 in.) and the distance from the tip of the nozzle to the first bend or point of disturbance shall be at least two times the outside nozzle diameter. The nozzle shall be constructed from seamless stainless steel tubing. Other configurations and construction material may be used with approval from the Administrator.

A range of sizes suitable for isokinetic sampling should be available, e.g., 0.32 cm (⅛ in.) up to 1.27 cm (½ in.) (or larger if higher volume sampling trains are used) inside diameter (ID) nozzles in increments of 0.16 cm ($\frac{1}{16}$ in.). Each nozzle shall be calibrated according to the procedures outlined in the calibration section.

5.1.2 Probe liner—Borosilicate glass or stainless steel (316). When the filter is located immediately after the probe, a probe heating system may be used to prevent filter plugging resulting from moisture condensation. The temperature in the probe shall not exceed 120 ± 14°C (248 ± 25°F).

5.1.3 Pitot tube—Type S, or other device approved by the Administrator, attached to probe to allow constant monitoring of the stack gas velocity. The face openings of the pitot tube and the probe nozzle shall be adjacent and parallel to each other, not necessarily on the same plane, during sampling. The free space between the nozzle and pitot tube shall be at least 1.9 cm (0.75 in.). The free space shall be set based on a 1.3 cm (0.5 in.) ID nozzle, which is the largest size nozzle used.

The pitot tube must also meet the criteria specified in Method 2 and be calibrated according to the procedure in the calibration section of that method.

5.1.4 Differential pressure gauge—Inclined manometer capable of measuring velocity head to within 10% of the minimum measured value. Below a differential pressure of 1.3 mm (0.05 in.) water gauge, micromanometers with sensitivities of 0.013 mm (0.0005 in.) should be used. However, micromanometers are not easily adaptable to field conditions and are not easy to use with pulsating flow. Thus, other methods or devices acceptable to the Administrator may be used when conditions warrant.

5.1.5 Filter holder—Borosilicate glass with a glass frit filter support and a silicone rubber gasket. Other materials of construction may be used with approval from the Administrator, e.g., if probe liner is stainless steel, then filter holder may be stainless steel. The holder design shall provide a positive seal against leakage from the outside or around the filter.

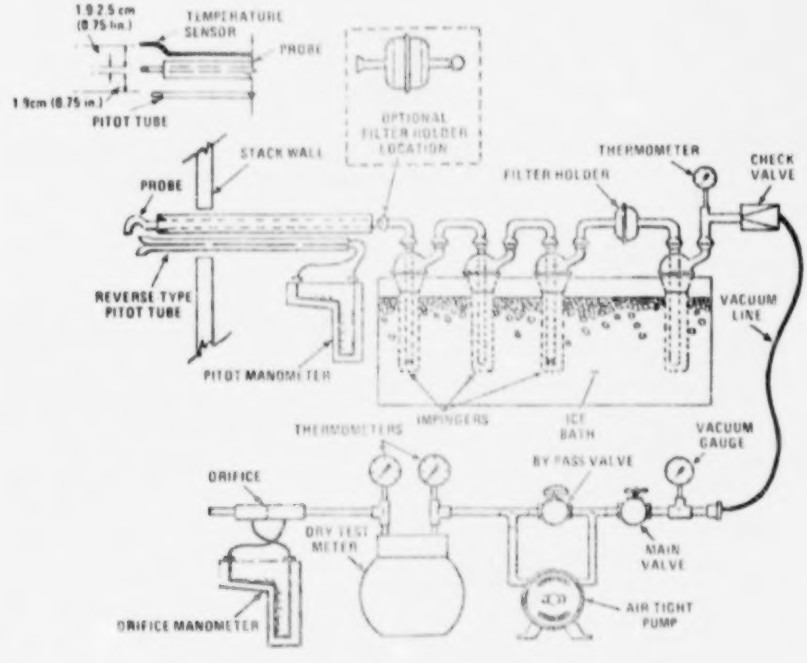

1.9-2.5 cm
(0.75 in.)
TEMPERATURE SENSOR
PROBE
1.9cm (0.75 in.)
PITOT TUBE
OPTIONAL FILTER HOLDER LOCATION
STACK WALL
PROBE
REVERSE TYPE PITOT TUBE
PITOT MANOMETER
THERMOMETER
CHECK VALVE
FILTER HOLDER
VACUUM LINE
IMPINGERS
ICE BATH
THERMOMETERS
VACUUM GAUGE
BY PASS VALVE
ORIFICE
DRY TEST METER
MAIN VALVE
AIR TIGHT PUMP
ORIFICE MANOMETER

Figure 13A-1. Fluoride sampling train.

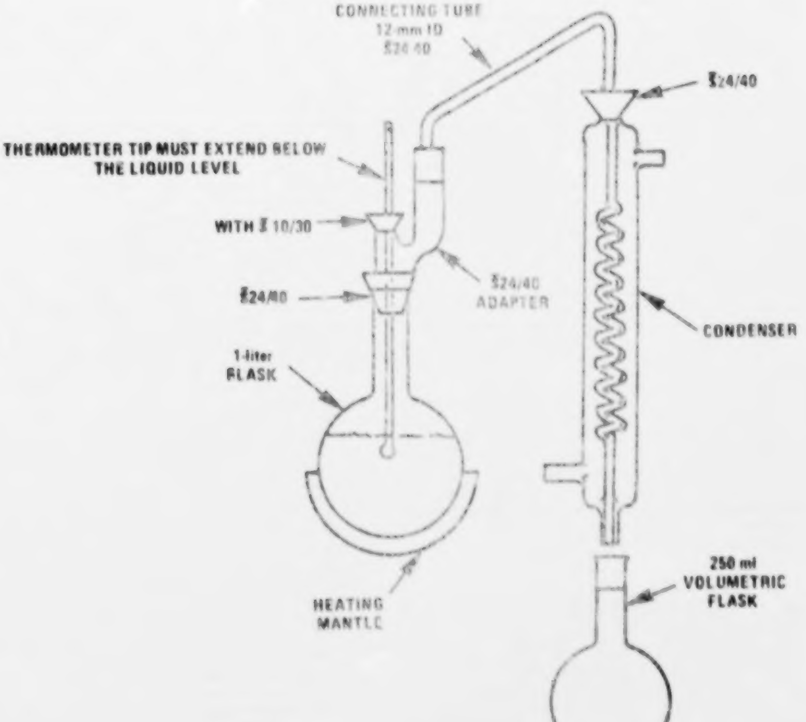

CONNECTING TUBE
12 mm ID
$24/40
$24/40
THERMOMETER TIP MUST EXTEND BELOW THE LIQUID LEVEL
WITH $ 10/30
$24/40
ADAPTER
$24/40
CONDENSER
1-liter FLASK
HEATING MANTLE
250 ml VOLUMETRIC FLASK

Figure 13A-2. Fluoride Distillation Apparatus

5.1.6 Filter heating system—When moisture condensation is a problem, any heating system capable of maintaining a temperature around the filter holder during sampling of no greater than 120 ± 14°C (248 ± 25°F). A temperature gauge capable of measuring temperature to within 3°C (5.4°F) shall be installed so that when the filter heater is used, the temperature around the filter holder can be regulated and monitored during sampling. Heating systems other than the one shown in APTD-0581 may be used.

5.1.7 Impingers—Four impingers connected as shown in Figure 13A-1 with ground glass (or equivalent), vacuum tight fittings. The first, third, and fourth impingers are of the Greenburg-Smith design, modified by replacing the tip with a 1¼ cm (½ in.) inside diameter glass tube extending to 1¼ cm (½ in.) from the bottom of the flask. The second impinger is of the Greenburg-Smith design with the standard tip.

5.1.8 Metering system—Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3°C (~5°F), dry gas meter with 2% accuracy at the required sampling rate, and related equipment, or equivalent, as required to maintain an isokinetic sampling rate and to determine sample volume. When the metering system is used in conjunction with a pitot tube, the system shall enable checks of isokinetic rates.

5.1.9 Barometer—Mercury, aneroid, or other barometers capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby weather bureau station, in which case the station value shall be requested and an adjustment for elevation differences shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase.

5.2 *Sample recovery.*

5.2.1 Probe liner and probe nozzle brushes—Nylon bristles with stainless steel wire handles. The probe brush shall have extensions, at least as long as the probe, of stainless steel, teflon, or similarly inert material. Both brushes shall be properly sized and shaped to brush out the probe liner and nozzle.

5.2.2 Glass wash bottles—Two.

5.2.3 Sample storage containers—Wide mouth, high density polyethylene bottles, 1 liter.

5.2.4 Plastic storage containers—Air tight containers of sufficient volume to store silica gel.

5.2.5 Graduated cylinder—250 ml.

5.2.6 Funnel and rubber policeman—to aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

5.3 *Analysis.*

5.3.1 Distillation apparatus—Glass distillation apparatus assembled as shown in Figure 13A-2.

5.3.2 Hot plate—Capable of heating to 500° C.

5.3.3 Electric muffle furnace—Capable of heating to 600° C.

5.3.4 Crucibles—Nickel, 75 to 100 ml capacity.

5.3.5 Beaker, 1500 ml.

5.3.6 Volumetric flask—50 ml.

5.3.7 Erlenmeyer flask or plastic bottle—500 ml.

5.3.8 Constant temperature bath—Capable of maintaining a constant temperature of ±1.0° C in the range of room temperature.

5.3.9 Balance—300 g capacity to measure to ±0.5 g.

5.3.10 Spectrophotometer — Instrument capable of measuring absorbance at 570 nm and providing at least a 1 cm light path.

5.3.11 Spectrophotometer cells—1 cm.

6. *Reagents*

6.1 *Sampling.*

6.1.1 Filters—Whatman No. 1 filters, or equivalent, sized to fit filter holder.

6.1.2 Silica gel—Indicating type, 6-16 mesh. If previously used, dry at 175° C (350° F) for 2 hours. New silica gel may be used as received.

6.1.3 Water—Distilled.

6.1.4 Crushed ice.

6.1.5 Stopcock grease—Acetone insoluble, heat stable silicone grease. This is not necessary if screw-on connectors with teflon sleeves, or similar, are used.

6.2 *Sample recovery.*

6.2.1 Water—Distilled from same container as 6.1.3.

6.3 *Analysis.*

6.3.1 Calcium oxide (CaO)—Certified grade containing 0.005 percent fluoride or less.

6.3.2 Phenolphthalein Indicator—0.1 percent in 1:1 ethanol-water mixture.

6.3.3 Silver sulfate ($Ag_2SO_4$)—ACS reagent grade, or equivalent.

6.3.4 Sodium hydroxide (NaOH)—Pellets, ACS reagent grade, or equivalent.

6.3.5 Sulfuric acid ($H_2SO_4$)—Concentrated, ACS reagent grade, or equivalent.

6.3.6 Filters—Whatman No. 541, or equivalent.

6.3.7 Hydrochloric acid (HCl)—Concentrated, ACS reagent grade, or equivalent.

6.3.8 Water—Distilled, from same container as 6.1.3.

6.3.9 Sodium fluoride—Standard solution. Dissolve 0.2210 g of sodium fluoride in 1 liter of distilled water. Dilute 100 ml of this solution to 1 liter with distilled water. One milliliter of the solution contains 0.01 mg of fluoride.

6.3.10 SPADNS solution—[4,5dihydroxy-3-(p-sulfophenylazo)-2,7-naphthalene-disulfonic acid trisodium salt]. Dissolve 0.960 ±.010 g of SPADNS reagent in 500 ml distilled water. This solution is stable for at least one month, if stored in a well-sealed bottle protected from sunlight.

6.3.11 Reference solution—Add 10 ml of SPADNS solution (6.3.10) to 100 ml distilled water and acidify with a solution prepared by diluting 7 ml of concentrated HCl to 10 ml with distilled water. This solution is used to set the spectrophotometer zero point and should be prepared daily.

6.3.12 SPADNS Mixed Reagent—Dissolve 0.135 ±0.005 g of zirconyl chloride octahydrate ($ZrOCl_2 \cdot 8H_2O$), in 25 ml distilled water. Add 350 ml of concentrated HCl and dilute to 500 ml with distilled water. Mix equal volumes of this solution and SPADNS solution to form a single reagent. This reagent is stable for at least two months.

7. *Procedure.*

NOTE: The fusion and distillation steps of this procedure will not be required, if it can be shown to the satisfaction of the Administrator that the samples contain only water-soluble fluorides.

7.1 *Sampling.* The sampling shall be conducted by competent personnel experienced with this test procedure.

7.1.1 Pretest preparation. All train components shall be maintained and calibrated according to the procedure described in APTD-0576, unless otherwise specified herein.

Weigh approximately 200-300 g of silica gel in air tight containers to the nearest 0.5 g. Record the total weight, both silica gel and container, on the container. More silica gel may be used but care should be taken during sampling that it is not entrained and carried out from the impinger. As an alternative, the silica gel may be weighed directly in the impinger or its sampling holder just prior to the train assembly.

7.1.2 Preliminary determinations. Select the sampling site and the minimum number of sampling points according to Method 1 or as specified by the Administrator. Determine the stack pressure, temperature, and the range of velocity heads using Method 2 and moisture content using Approximation Method 4 or its alternatives for the purpose of making isokinetic sampling rate calculations. Estimates may be used. However, final results will be based on actual measurements made during the test.

Select a nozzle size based on the range of velocity heads such that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle size. Ensure that the differential pressure gauge is capable of measuring the minimum velocity head value to within 10%, or as specified by the Administrator.

Select a suitable probe liner and probe length such that all traverse points can be sampled. Consider sampling from opposite sides for large stacks to reduce the length of probes.

Select a total sampling time greater than or equal to the minimum total sampling time specified in the test procedures for the specific industry such that the sampling time per point is not less than 2 min. or select some greater time interval as specified by the Administrator, and such that the sample volume that will be taken will exceed the required minimum total gas sample volume specified in the test procedures for the specific industry. The latter is based on an approximate average sampling rate. Note also that the minimum total sample volume is corrected to standard conditions.

It is recommended that a half-integral or integral number of minutes be sampled at each point in order to avoid timekeeping errors.

In some circumstances, e.g. batch cycles, it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes. In these cases, the Administrator's approval must first be obtained.

7.1.3 Preparation of collection train. During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to begin.

Place 100 ml of water in each of the first two impingers, leave the third impinger empty, and place approximately 200-300 g or more, if necessary, of preweighed silica gel in the fourth impinger. Record the weight of the silica gel and container on the data sheet. Place the empty container in a clean place for later use in the sample recovery.

Place a filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as to not allow the sample gas stream to circumvent the filter. Check filter for tears after assembly is completed.

When glass liners are used, install selected nozzle using a Viton A O-ring; the Viton A O-ring is installed as a seal where the nozzle is connected to a glass liner. See APTD-0576 for details. When metal liners are used, install the nozzle as above or by a leak free direct mechanical connection. Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

Unless otherwise specified by the Administrator, attach a temperature probe to the metal sheath of the sampling probe so that the sensor extends beyond the probe tip and does not touch any metal. Its position should be about 1.9 to 2.54 cm (0.75 to 1 in.) from the pitot tube and probe nozzle to avoid interference with the gas flow.

Assemble the train as shown in Figure 13A-1 with the filter between the third and fourth impingers. Alternatively, the filter may be placed between the probe and the first impinger. A filter heating system may be used to prevent moisture condensation, but the temperature around the filter holder shall not exceed 120±14°C (248±25°F). [(Note: Whatman No. 1 filter decomposes at 150°C (300°F).)] Record filter location on the data sheet.

Place crushed ice around the impingers.

7.1.4 Leak check procedure—After the sampling train has been assembled, turn on and set (if applicable) the probe and filter heating system(s) to reach a temperature sufficient to avoid condensation in the probe. Allow time for the temperature to stabilize. Leak check the train at the sampling site by plugging the nozzle and pulling a 380 mm Hg (15 in. Hg) vacuum. A leakage rate in excess of 4% of the average sampling rate or 0.00057 $m^3$/min (0.02 cfm), whichever is less, is unacceptable.

The following leak check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with by-pass valve fully open and coarse adjust valve completely closed. Partially open the coarse adjust valve and slowly close the by-pass valve until 380 mm Hg (15 in. Hg) vacuum is reached. *Do not* reverse direction of by-pass valve. This will cause water to back up into the filter holder. If 380 mm Hg (15 in. Hg) is exceeded, either leak check at this higher vacuum or end the leak check as described below and start over.

When the leak check is completed, first slowly remove the plug from the inlet to the probe or filter holder and immediately turn off the vacuum pump. This prevents the water in the impingers from being forced backward into the filter holder (if placed before the impingers) and silica gel from being entrained backward into the third impinger.

Leak checks shall be conducted as described whenever the train is disengaged, e.g. for silica gel or filter changes during the test, prior to each test run, and at the completion of each test run. If leaks are found to be in excess of the acceptable rate, the test will be considered invalid. To reduce lost time due to leakage occurrences, it is recommended that leak checks be conducted between port changes.

7.1.5 Particulate train operation—During the sampling run, an isokinetic sampling rate within 10%, or as specified by the Administrator, of true isokinetic shall be maintained.

For each run, record the data required on the example data sheet shown in Figure 13A-3. Be sure to record the initial dry gas meter reading. Record the dry gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made, and when sampling is halted. Take other data point readings at least once at each sample point during each time increment and additional readings when significant changes (20% variation in velocity head readings) necessitate additional adjustments in flow rate. Be sure to level and zero the manometer.

Clean the portholes prior to the test run to minimize chance of sampling deposited material. To begin sampling, remove the nozzle cap, verify (if applicable) that the probe heater is working and filter heater is up to temperature, and that the pitot tube and probe are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs are available for sampling trains using type S pitot tubes with 0.85±0.02 coefficients ($C_p$), and when sampling in air or a stack gas with equivalent density (molecular weight $M_d$, equal to 29 ± 4), which aid in the rapid adjustment of the isokinetic sampling rate without excessive computations. APTD-0576 details the procedure for using these nomographs. If $C_p$ and $M_d$ are outside the above stated ranges, do not use the nomograph unless appropriate steps are taken to compensate for the deviations.

When the stack is under significant negative pressure (height of impinger stem), take care to close the coarse adjust valve before inserting the probe into the stack to avoid water backing into the filter holder. If necessary, the pump may be turned on with the coarse adjust valve closed.

When the probe is in position, block off the openings around the probe and porthole to prevent unrepresentative dilution of the gas stream.

Traverse the stack cross section, as required by Method 1 or as specified by the Administrator, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the portholes to minimize chance of extracting deposited material.

During the test run, make periodic adjustments to keep the probe and (if applicable) filter temperatures at their proper values. Add more ice and, if necessary, salt to the ice bath, to maintain a temperature of less than 20°C (68°F) at the impinger/silica gel outlet, to avoid excessive moisture losses. Also, periodically check the level and zero of the manometer.

If the pressure drop across the filter becomes high enough to make isokinetic sampling difficult to maintain, the filter may be replaced in the midst of a sample run. It is recommended that another complete filter assembly be used rather than attempting to change the filter itself. After the new filter or filter assembly is installed conduct a leak check. The final emission results shall be based on the summation of all filter catches.

A single train shall be used for the entire sample run, except for filter and silica gel changes. However, if approved by the Administrator, two or more trains may be used for a single test run when there are two or more ducts or sampling ports. The final emission results shall be based on the total of all sampling train catches.

At the end of the sample run, turn off the pump, remove the probe and nozzle from the stack, and record the final dry gas meter reading. Perform a leak check.[1] Calculate percent isokinetic (see calculation section) to determine whether another test run should be made. If there is difficulty in maintaining isokinetic rates due to source conditions, consult with the Administrator for possible variance on the isokinetic rates.

7.2 Sample recovery. Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period.

When the probe can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it to keep from losing part of the sample. Do not cap off the probe tip tightly while the sampling train is cooling down, as this would create a vacuum in the filter holder, thus drawing water from the impingers into the filter.

Before moving the sample train to the cleanup site, remove the probe from the sample train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate, if present. Wipe off the silicone grease from the filter inlet where the probe was fastened and cap it. Remove the umbilical cord from the last impinger and cap the impinger. After wiping off the silicone grease, cap off the filter holder outlet and impinger inlet. Ground glass stoppers, plastic caps, or serum caps may be used to close these openings.

Transfer the probe and filter-impinger assembly to the cleanup area. This area should be clean and protected from the wind so that the chances of contaminating or losing the sample will be minimized.

Inspect the train prior to and during disassembly and note any abnormal conditions. Using a graduated cylinder, measure and record the volume of the water in the first three impingers, to the nearest ml; any condensate in the probe should be included in this determination. Treat the samples as follows:

7.2.1 Container No. 1. Transfer the impinger water from the graduated cylinder to this container. Add the filter to this container. Wash all sample exposed surfaces, including the probe tip, probe, first three impingers, impinger connectors, filter holder, and graduated cylinder thoroughly with distilled water. Wash each component three separate times with water and clean the probe and nozzle with brushes. A maximum wash of 500 ml is used, and the washings are added to the sample container which must be made of polyethylene.

7.2.2 Container No. 2. Transfer the silica gel from the fourth impinger to this container and seal.

7.3 *Analysis.* Treat the contents of each sample container as described below.

7.3.1 Container No. 1.

7.3.1.1 Filter this container's contents, including the Whatman No. 1 filter, through Whatman No. 541 filter paper, or equivalent into a 1500 ml beaker. Note: If filtrate volume exceeds 900 ml make filtrate basic with NaOH to phenolphthalein and evaporate to less than 900 ml.

7.3.1.2 Place the Whatman No. 541 filter containing the insoluble matter (including the Whatman No. 1 filter) in a nickel crucible, add a few ml of water and macerate the filter with a glass rod.

Add 100 mg CaO to the crucible and mix the contents thoroughly to form a slurry. Add a couple of drops of phenolphthalein indicator. The indicator will turn red in a basic medium. The slurry should remain basic during the evaporation of the water or fluoride ion will be lost. If the indicator turns colorless during the evaporation, an acidic condition is indicated. If this happens add CaO until the color turns red again.

Place the crucible in a hood under infrared lamps or on a hot plate at low heat. Evaporate the water completely.

After evaporation of the water, place the crucible on a hot plate under a hood and slowly increase the temperature until the paper chars. It may take several hours for complete charring of the filter to occur.

Place the crucible in a cold muffle furnace and gradually (to prevent smoking) increase the temperature to 600°C, and maintain until the contents are reduced to an ash. Remove the crucible from the furnace and allow it to cool.

7.3.1.3 Add approximately 4 g of crushed NaOH to the crucible and mix. Return the crucible to the muffle furnace, and fuse the sample for 10 minutes at 600°C.

Remove the sample from the furnace and cool to ambient temperature. Using several rinsings of warm distilled water transfer the contents of the crucible to the beaker containing the filtrate from container No. 1 (7.3.1). To assure complete sample removal, rinse finally with two 20 ml portions of 25 percent (v/v) sulfuric acid and carefully add to the beaker. Mix well and transfer to one-liter volumetric flask. Dilute to volume with distilled water and mix thoroughly. Allow any undissolved solids to settle.

---

[1] With acceptability of the test run to be based on the same criterion as in 7.1.4.

7.3.2 Container No. 2. Weigh the spent silica gel and report to the nearest 0.5 g.

7.3.3 Adjustment of acid/water ratio in distillation flask—(Utilize a protective shield when carrying out this procedure.) Place 400 ml of distilled water in the distilling flask and add 200 ml of concentrated $H_2SO_4$. Caution: Observe standard precautions when mixing the $H_2SO_4$ by slowly adding the acid to the flask with constant swirling. Add some soft glass beads and several small pieces of broken glass tubing and assemble the apparatus as shown in Figure 13A-2. Heat the flask until it reaches a temperature of 175° C to adjust the acid/water ratio for subsequent distillations. Discard the distillate.

7.3.4 Distillation—Cool the contents of the distillation flask to below 80° C. Pipette an aliquot of sample containing less than 0.6 mg F directly into the distilling flask and add distilled water to make a total volume of 220 ml added to the distilling flask. [For an estimate of what size aliquot does not exceed 0.6 mg F, select an aliquot of the solution and treat as described in Section 7.3.6. This will give an approximation of the fluoride content, but only an approximation since interfering ions have not been removed by the distillation step.]

Place a 250 ml volumetric flask at the condenser exit. Now begin distillation and gradually increase the heat and collect all the distillation up to 175°C. Caution: Heating the solution above 175°C will cause sulfuric acid to distill over.

The acid in the distilling flask can be used until there is carryover of interferences or poor fluoride recovery. An occasional check of fluoride recovery with standard solutions is advised. The acid should be changed whenever there is less than 90 percent recovery or blank values are higher than 0.1 µg/ml. Note: If the sample contains chloride, add 5 mg $Ag_2SO_4$ to the flask for every mg of chloride. Gradually increase the heat and collect all the distillate up to 175° C. Do not exceed 175°C.

7.3.5 Determination of Concentration—Bring the distillate in the 250 ml volumetric flask to the mark with distilled water and mix thoroughly. Pipette a suitable aliquot from the distillate (containing 10 µg to 40 µg fluoride) and dilute to 50 ml with distilled water. Add 10 ml of SPADNS Mixed Reagent (see Section 6.3.12) and mix thoroughly.

After mixing, place the sample in a constant temperature bath containing the standard solution for thirty minutes before reading the absorbance with the spectrophotometer.

Set the spectrophotometer to zero absorbance at 570 nm with reference solution (6.3.11), and check the spectrophotometer calibration with the standard solution. Determine the absorbance of the samples and determine the concentration from the calibration curve. If the concentration does not fall within the range of the calibration curve, repeat the procedure using a different size aliquot.

8. *Calibration.*

Maintain a laboratory log of all calibrations.

8.1 Sampling Train.

8.1.1 Probe nozzle—Using a micrometer, measure the inside diameter of the nozzle to the nearest 0.025 mm (0.001 in.). Make 3 separate measurements using different diameters each time and obtain the average of the measurements. The difference between the high and low numbers shall not exceed 0.1 mm (0.004 in.).

When nozzles become nicked, dented, or corroded, they shall be reshaped, sharpened, and recalibrated before use.

Each nozzle shall be permanently and uniquely identified.

8.1.2 Pitot tube—The pitot tube shall be calibrated according to the procedure outlined in Method 2.

8.1.3 Dry gas meter and orifice meter. Both meters shall be calibrated according to the procedure outlined in APTD-0576. When diaphragm pumps with by-pass valves are used, check for proper metering system design by calibrating the dry gas meter at an additional flow rate of 0.0057 m³/min. (0.2 cfm) with the by-pass valve fully opened and then with it fully closed. If there is more than ±2 percent difference in flow rates when compared to the fully closed position of the by-pass valve, the system is not designed properly and must be corrected.

8.1.4 Probe heater calibration—The probe heating system shall be calibrated according to the procedure contained in APTD-0576. Probes constructed according to APTD-0581 need not be calibrated if the calibration curves in APTD-0576 are used.

8.1.5 Temperature gauges—Calibrate dial and liquid filled bulb thermometers against mercury-in-glass thermometers. Thermocouples need not be calibrated. For other devices, check with the Administrator.

8.2 Analytical Apparatus. Spectrophotometer. Prepare the blank standard by adding 10 ml of SPADNS mixed reagent to 50 ml of distilled water. Accurately prepare a series of standards from the standard fluoride solution (see Section 6.3.9) by diluting 2, 4, 6, 8, 10, 12, and 14 ml volumes to 100 ml with distilled water. Pipette 50 ml from each solution and transfer to a 100 ml beaker. Then add 10 ml of SPADNS mixed reagent to each. These standards will contain 0, 10, 20, 30, 40, 50, 60, and 70 µg of fluoride (0—1.4 µg/ml) respectively.

After mixing, place the reference standards and reference solution in a constant temperature bath for thirty minutes before reading the absorbance with the spectrophotometer. All samples should be adjusted to this same temperature before analyzing. Since a 3°C temperature difference between samples and standards will produce an error of approximately 0.005 mg F/liter, care must be taken to see that samples and standards are at nearly identical temperatures when absorbances are recorded.

With the spectrophotometer at 570 nm, use the reference solution (see section 6.3.11) to set the absorbance to zero.

Determine the absorbance of the standards. Prepare a calibration curve by plotting µg F/50 ml versus absorbance on linear graph paper. A standard curve should be prepared initially and thereafter whenever the SPADNS mixed reagent is newly made. Also, a calibration standard should be run with each set of samples and if it differs from the calibration curve by ±2 percent, a new standard curve should be prepared.

9. *Calculations.*

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

9.1 Nomenclature.

$A_d$ = Aliquot of distillate taken for color development, ml.

$A_n$ = Cross sectional area of nozzle, m² (ft²).

$A_t$ = Aliquot of total sample added to still, ml.

$B_{ws}$ = Water vapor in the gas stream, proportion by volume.

$C_s$ = Concentration of fluoride in stack gas, mg/m³, corrected to standard conditions of 20° C, 760 mm Hg (68° F, 29.92 in. Hg) on dry basis.
$F_t$ = Total weight of fluoride in sample, mg.
$\mu gF$ = Concentration from the calibration curve, μg.
$I$ = Percent of isokinetic sampling.
$m_n$ = Total amount of particulate matter collected, mg.
$M_w$ = Molecular weight of water, 18 g/g-mole (18 lb/lb-mole).
$m_a$ = Mass of residue of acetone after evaporation, mg.
$P_{bar}$ = Barometric pressure at the sampling site, mm Hg (in. Hg).
$P_s$ = Absolute stack gas pressure, mm Hg (in. Hg).
$P_{std}$ = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).
$R$ = Ideal gas constant, 0.06236 mm Hg-m³/°K-g-mole (21.83 in. Hg-ft³/°R-lb-mole).
$T_m$ = Absolute average dry gas meter temperature (see fig. 13A-3), °K (°R).
$T_s$ = Absolute average stack gas temperature (see fig. 13A-3), °K (°R).
$T_{std}$ = Standard absolute temperature, 293° K (528° R).
$V_a$ = Volume of acetone blank, ml.
$V_{aw}$ = Volume of acetone used in wash, ml.
$V_d$ = Volume of distillate collected, ml.
$V_{lc}$ = Total volume of liquid collected in impingers and silica gel, ml. Volume of water in silica gel equals silica gel weight increase in grams times 1 ml/gram. Volume of liquid collected in impinger equals final volume minus initial volume.
$V_m$ = Volume of gas sample as measured by dry gas meter, dcm (dcf).
$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter corrected to standard conditions, dscm (dscf).
$V_{w(std)}$ = Volume of water vapor in the gas sample corrected to standard conditions, scm (scf).
$V_t$ = Total volume of sample, ml.
$v_s$ = Stack gas velocity, calculated by Method 2, Equation 2-7 using data obtained from Method 5, m/sec (ft/sec).
$W_a$ = Weight of residue in acetone wash, mg.
$\Delta H$ = Average pressure differential across the orifice (see fig. 13A-3), meter, mm H₂O (in. H₂O).
$\rho_a$ = Density of acetone, mg/ml (see label on bottle).
$\rho_w$ = Density of water, 1 g/ml (0.00220 lb/ml).
$\theta$ = Total sampling time, min.
13.6 = Specific gravity of mercury.
60 = Sec/min.
100 = Conversion to percent.

9.2 Average dry gas meter temperature and average orifice pressure drop. See data sheet (fig. 13A-3).

9.3 Dry gas volume. Correct the sample volume measured by the dry gas meter to standard conditions [20° C, 760 mm Hg (68° F, 29.92 inches Hg)] by using equation 13A-1.

$$V_{m(std)} = V_m \frac{T_{std}}{T_m}\left[\frac{P_{bar}+\frac{\Delta H}{13.6}}{P_{std}}\right] = K V_m \frac{P_{bar}+\Delta H/13.6}{T_m}$$

equation 13A-1

where:
$K$ = 0.3855 °K/mm Hg for metric units.
= 17.65 °R/in. Hg for English units.

9.4 Volume of water vapor.

$$V_{w(std)} = V_{lc} \frac{\rho_w}{M_w} \frac{R T_{std}}{P_{std}} = K V_{lc}$$

equation 13A-2

where:
$K$ = 0.00134 m³/ml for metric units.
= 0.0472 ft³/ml for English units.

9.5 Moisture content.

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}}$$

equation 13A-3

If the liquid droplets are present in the gas stream assume the stream to be saturated and use a psychrometric chart to obtain an approximation of the moisture percentage.

9.6 Concentration.

9.6.1 Calculate the amount of fluoride in the sample according to Equation 13A-4.

$$F_t = K \frac{V_t}{A_t} \frac{V_d}{A_d} (\mu g\, F)$$

equation 13A-4

where:
$K = 10^{-3}$ mg/μg.

9.6.2 Concentration of fluoride in stack gas. Determine the concentration of fluoride in the stack gas according to Equation 13A-5.

$$C_s = K \frac{F_t}{V_{m(std)}}$$

equation 13A-5

where:
$K$ = 35.31 ft³/m³.

9.7 Isokinetic variation.

9.7.1 Calculations from raw data.

$$I=\frac{100\,T_s\,[KV_{lc}+(V_m/T_m)\,(P_{bar}+\Delta H/13.6)]}{60\,\theta v_s P_s A_n} \qquad \text{equation 13A-6}$$

where:

$K=0.00346$ mm Hg-m³/ml-°K for metric units.

$=0.00267$ in. Hg-ft³/ml-°R for English units.

9.7.2 Calculations from intermediate values.

$$I=\frac{T_s V_{m(std)} P_{std}\,100}{T_{std} v_s \theta A_n P_s\,60\,(1-B_{ws})}$$

$$=K\,\frac{T_s V_{m(std)}}{P_s v_s A_n \theta\,(1-B_{ws})} \qquad \text{equation 13A-7}$$

where:

$K=4.323$ for metric units.

$=0.0944$ for English units.

9.8 Acceptable results. The following range sets the limit on acceptable isokinetic sampling results:

If 90 percent $<I<$ 110 percent, the results are acceptable. If the results are low in comparison to the standards and I is beyond the acceptable range, the Administrator may option to accept the results. Use reference 7.4 to make judgments. Otherwise, reject the results and repeat the test.

10. *References.*

Bellack, Ervin, "Simplified Fluoride Distillation Method," *Journal of the American Water Works Association* #50: 530-6 (1958).

MacLeod, Kathryn E., and Howard L. Crist, "Comparison of the SPADNS—Zirconium Lake and Specific Ion Electrode Methods of Fluoride Determination in Stack Emission Samples," *Analytical Chemistry* 45: 1272-1273 (1973).

Martin, Robert M., "Construction Details of Isokinetic Source Sampling Equipment," Environmental Protection Agency, Air Pollution Control Office Publication No. APTD-0581.

*1973 Annual Book of ASTM Standards*, Part 23, Designation: D 1179-72.

Rom, Jerome J., "Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment," Environmental Protection Agency, Air Pollution Control Office Publication No. APTD-0576.

*Standard Methods for the Examination of Water and Waste Water*, published jointly by American Public Health Association, American Water Works Association and Water Pollution Control Federation, 13th Edition (1971).

PLANT ____

LOCATION ____

OPERATOR ____

DATE ____

RUN NO. ____

SAMPLE BOX NO. ____

METER BOX NO. ____

METER ΔH@ ____

C FACTOR ____

PITOT TUBE COEFFICIENT $C_p$ ____

SCHEMATIC OF STACK CROSS SECTION

AMBIENT TEMPERATURE ____

BAROMETRIC PRESSURE ____

ASSUMED MOISTURE, % ____

PROBE LENGTH, m (ft) ____

NOZZLE IDENTIFICATION NO. ____

AVERAGE CALIBRATED NOZZLE DIAMETER, cm (in.) ____

PROBE HEATER SETTING ____

LEAK RATE, m³/min (cfm) ____

PROBE LINER MATERIAL ____

| TRAVERSE POINT NUMBER | SAMPLING TIME (θ) min | STATIC PRESSURE mm Hg (in. Hg) | STACK TEMPERATURE ($T_S$) °C (°F) | VELOCITY HEAD ($\Delta P_S$) | PRESSURE DIFFERENTIAL ACROSS ORIFICE METER mm $H_2O$ (in. $H_2O$) | GAS SAMPLE VOLUME m³ (ft³) | GAS SAMPLE TEMPERATURE AT DRY GAS METER | | FILTER HOLDER TEMPERATURE °C (°F) | TEMPERATURE OF GAS LEAVING CONDENSER OR LAST IMPINGER °C (°F) |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | INLET °C (°F) | OUTLET °C (°F) | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| TOTAL | | | | | | | Avg. | Avg. | | |
| AVERAGE | | | | | | | Avg. | | | |

Figure 13A-3. Field data.

Reference Method 13A is amended as follows:

(a) In section 3., the phrase "300 μg/liter" is corrected to read "300 mg/liter" and the parenthetical phrase "(see section 7.3.6)" is corrected to read "(see section 7.3.4)".

(b) Section 5.1.5 is revised to read as follows:

5.1.5 Filter holder—If located between the probe and first impinger, borosilicate glass with a 20 mesh stainless steel screen filter support and a silicone rubber gasket: neither a glass frit filter support nor a sintered metal filter support may be used if the filter is in front of the impingers. If located between the third and fourth impingers, borosilicate glass with a glass frit filter support and a silicone rubber gasket. Other materials of construction may be used with approval from the Administrator, e.g., if probe liner is stainless steel, then filter holder may be stainless steel. The holder design shall provide a positive seal against leakage from the outside or around the filter.

(c) Section 7.1.3 is amended by revising the first two sentences of the sixth paragraph to read as follows:

7.1.3 Preparation of collection train. * * * Assemble the train as shown in Figure 13A-1 with the filter between the third and fourth impingers. Alternatively, the filter may be placed between the probe and first impinger if a 20 mesh stainless steel screen is used for the filter support. * * *

* * * * *

(d) In section 7.3.4, the reference in the first paragraph to "section 7.3.6" is corrected to read "section 7.3.5".

# METHOD 13B

# DETERMINATION OF TOTAL FLUORIDE EMISSIONS FROM STATIONARY SOURCES - SPECIFIC ION ELECTRODE METHOD

1. *Principle and Applicability.*

1.1 Principle. Gaseous and particulate fluorides are withdrawn isokinetically from the source using a sampling train. The fluorides are collected in the impinger water and on the filter of the sampling train. The weight of total fluorides in the train is determined by the specific ion electrode method.

1.2 Applicability. This method is applicable for the determination of fluoride emissions from stationary sources only when specified by the test procedures for determining compliance with new source performance standards. Fluorocarbons such as Freons, are not quantitatively collected or measured by this procedure.

2. *Range and Sensitivity.*

The fluoride specific ion electrode analytical method covers the range of 0.02–2,000 μg F/ml; however, measurements of less than 0.1 μg F/ml require extra care. Sensitivity has not been determined.

3. *Interferences.*

During the laboratory analysis, aluminum in excess of 300 mg/liter and silicon dioxide in excess of 300 μg/liter will prevent complete recovery of fluoride.

4. *Precision, Accuracy and Stability.*

The accuracy of fluoride electrode measurements has been reported by various researchers to be in the range of 1–5 percent in a concentration range of 0.04 to 80 mg/l. A change in the temperature of the sample will change the electrode response; a change of 1°C will produce a 1.5 percent relative error in the measurement. Lack of stability in the electrometer used to measure EMF can introduce error. An error of 1 millivolt in the EMF measurement produces a relative error of 4 percent regardless of the absolute concentration being measured.

5. *Apparatus.*

5.1 Sample train. See Figure 13A-1 (Method 13A); it is similar to the Method 5 train except for the interchangeability of the position of the filter. Commercial models of this train are available. However, if one desires to build his own, complete construction details are described in APTD-0581; for changes from the APTD-0581 document and for allowable modifications to Figure 13A-1, see the following subsections.

The operating and maintenance procedures for the sampling train are described in APTD-0576. Since correct usage is important in obtaining valid results, all users should read the APTD-0576 document and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein.

5.1.1 Probe nozzle—Stainless steel (316) with sharp, tapered leading edge. The angle of taper shall be ≤30° and the taper shall be on the outside to preserve a constant internal diameter. The probe nozzle shall be of the button-hook or elbow design, unless otherwise specified by the Administrator. The wall thickness of the nozzle shall be less than or equal to that of 20 gauge tubing, i.e., 0.165 cm (0.065 in.) and the distance from the tip of the nozzle to the first bend or point of disturbance shall be at least two times the outside nozzle diameter. The nozzle shall be constructed from seamless stainless steel tubing. Other configurations and construction material may be used with approval from the Administrator.

A range of sizes suitable for isokinetic sampling should be available, e.g., 0.32 cm ($\frac{1}{8}$ in.) up to 1.27 cm ($\frac{1}{2}$ in.) (or larger if higher volume sampling trains are used) inside diameter (ID) nozzles in increments of 0.16 cm ($\frac{1}{16}$ in.). Each nozzle shall be calibrated according to the procedures outlined in the calibration section.

5.1.2 Probe liner—Borosilicate glass or stainless steel (316). When the filter is located immediately after the probe, a probe heating system may be used to prevent filter plugging resulting from moisture condensation. The temperature in the probe shall not exceed 120±14°C (248±25°F).

5.1.3 Pitot tube—Type S, or other device approved by the Administrator, attached to probe to allow constant monitoring of the stack gas velocity. The face openings of the pitot tube and the probe nozzle shall be adjacent and parallel to each other, not necessarily on the same plane, during sampling. The free space between the nozzle and pitot tube shall be at least 1.9 cm (0.75 in.). The free space shall be set based on a 1.3 cm (0.5 in.) ID nozzle, which is the largest size nozzle used.

The pitot tube must also meet the criteria specified in Method 2 and be calibrated according to the procedure in the calibration section of that method.

5.1.4 Differential pressure gauge—Inclined manometer capable of measuring velocity head to within 10 percent of the minimum measured value. Below a differential pressure of 1.3 mm (0.05 in.) water gauge, micromanometers with sensitivities of 0.013 mm (0.0005 in.) should be used. However, micromanometers are not easily adaptable to field conditions and are not easy to use with pulsating flow. Thus, other methods or devices acceptable to the Administrator may be used when conditions warrant.

5.1.5 Filter holder—Borosilicate glass with a glass frit filter support and a silicone rubber gasket. Other materials of construction may be used with approval from the Administrator, e.g. if probe liner is stainless steel, then filter holder may be stainless steel. The holder design shall provide a positive seal against leakage from the outside or around the filter.

5.1.6 Filter heating system—When moisture condensation is a problem, any heating system capable of maintaining a temperature around the filter holder during sampling of no greater than 120±14°C (248±25°F). A temperature gauge capable of measuring temperature to within 3°C (5.4°F) shall be installed so that when the filter heater is used, the temperature around the filter holder can be regulated and monitored during sampling.

Heating systems other than the one shown in APTD-0581 may be used.

5.1.7 Impingers—Four impingers connected as shown in Figure 13A-1 with ground glass (or equivalent) vacuum tight fittings. The first, third, and fourth impingers are of the Greenburg-Smith design, modified by replacing the tip with a 1¼ cm (½ in.) inside diameter glass tube extending to 1¼ cm (½ in.) from the bottom of the flask. The second impinger is of the Greenburg-Smith design with the standard tip.

5.1.8 Metering system—Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3°C (±5°F), dry gas meter with 2 percent accuracy at the required sampling rate, and related equipment, or equivalent, as required to maintain an isokinetic sampling rate and to determine sample volume. When the metering system is used in conjunction with a pitot tube, the system shall enable checks of isokinetic rates.

5.1.9 Barometer—Mercury, aneroid, or other barometers capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in Hg). In many cases, the barometric reading may be obtained from a nearby weather bureau station, in which case the station value shall be requested and an adjustment for elevation differences shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase.

5.2 Sample recovery.

5.2.1 Probe liner and probe nozzle brushes—Nylon bristles with stainless steel wire handles. The probe brush shall have extensions, at least as long as the probe, of stainless steel, teflon, or similarly inert material. Both brushes shall be properly sized and shaped to brush out the probe liner and nozzle.

5.2.2 Glass wash bottles—Two.

5.2.3 Sample storage containers—Wide mouth, high density polyethylene bottles, 1 liter.

5.2.4 Plastic storage containers—Air tight containers of sufficient volume to store silica gel.

5.2.5 Graduated cylinder—250 ml.

5.2.6 Funnel and rubber policeman—To aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

5.3 Analysis.

5.3.1 Distillation apparatus—Glass distillation apparatus assembled as shown in Figure 13A-2 (Method 13A).

5.3.2 Hot plate—Capable of heating to 500°C.

5.3.3 Electric muffle furnace—Capable of heating to 600°C.

5.3.4 Crucibles—Nickel, 75 to 100 ml capacity.

5.3.5 Beaker—1500 ml.

5.3.6 Volumetric flask—50 ml.

5.3.7 Erlenmeyer flask or plastic bottle—500 ml.

5.3.8 Constant temperature bath—Capable of maintaining a constant temperature of ±1.0°C in the range of room temperature.

5.3.9 Trip balance—300 g capacity to measure to ±0.5 g.

5.3.10 Fluoride ion activity sensing electrode.

5.3.11 Reference electrode—Single junction; sleeve type. (A combination-type electrode having the references electrode and the fluoride-ion sensing electrode built into one unit may also be used).

5.3.12 Electrometer—A pH meter with millivolt scale capable of ±0.1 mv resolution, or a specific ion meter made specifically for specific ion use.

5.3.13 Magnetic stirrer and TFE fluorocarbon coated stripping bars.

6. *Reagents.*

6.1 Sampling.

6.1.1 Filters—Whatman No. 1 filters, or equivalent, sized to fit filter holder.

6.1.2 Silica gel—Indicating type, 6-16 mesh. If previously used, dry at 175°C (350°F) for 2 hours. New silica gel may be used as received.

6.1.3 Water—Distilled.

6.1.4 Crushed ice.

6.1.5 Stopcock grease—Acetone insoluble, heat stable silicone grease. This is not necessary if screw-on connectors with teflon sleeves, or similar, are used.

6.2 Sample recovery.

6.2.1 Water—Distilled from same container as 6.1.3

6.3 Analysis.

6.3.1 Calcium oxide (CaO)—Certified grade containing 0.005 percent fluoride or less.

6.3.2 Phenolphthalein Indicator—0.1 percent in 1:1 ethanol water mixture.

6.3.3 Sodium hydroxide (NaOH)—Pellets, ACS reagent grade or equivalent.

6.3.4 Sulfuric acid ($H_2SO_4$)—Concentrated, ACS reagent grade or equivalent.

6.3.5 Filters—Whatman No. 541, or equivalent.

6.3.6 Water—Distilled, from same container as 6.1.3.

6.3.7 Total Ionic Strength Adjustment Buffer (TISAB)—Place approximately 500 ml of distilled water in a 1-liter beaker. Add 57 ml glacial acetic acid, 58 g sodium chloride, and 4 g CDTA (Cyclohexylene dinitrilo tetraacetic acid). Stir to dissolve. Place the beaker in a water bath to cool it. Slowly add 5 M NaOH to the solution, measuring the pH continuously with a calibrated pH/reference electrode pair, until the pH is 5.3. Cool to room temperature. Pour into a 1-liter flask and dilute to volume with distilled water. Commercially prepared TISAB buffer may be substituted for the above.

6.3.8 Fluoride Standard Solution—0.1 M fluoride reference solution. Add 4.20 grams of reagent grade sodium fluoride (NaF) to a 1-liter volumetric flask and add enough distilled water to dissolve. Dilute to volume with distilled water.

7. *Procedure.*

NOTE: The fusion and distillation steps of this procedure will not be required, if it can be shown to the satisfaction of the Administrator that the samples contain only water-soluble fluorides.

7.1 Sampling. The sampling shall be conducted by competent personnel experienced with this test procedure.

7.1.1 Pretest preparation. All train components shall be maintained and calibrated according to the procedure described in APTD-0576, unless otherwise specified herein.

Weigh approximately 200-300 g of silica gel in air tight containers to the nearest 0.5 g. Record the total weight, both silica gel and container, on the container. More silica gel may be used but care should be taken during sampling that it is not entrained and carried out from the impinger. As an alternative, the silica gel may be weighed directly in the impinger or its sampling holder just prior to the train assembly.

7.1.2 Preliminary determinations. Select the sampling site and the minimum number of sampling points according to Method 1 or as specified by the Administrator. Determine the stack pressure, temperature, and the range of velocity heads using Method 2 and moisture content using Approximation Method 4 or its alternatives for the purpose of making isokinetic sampling rate calculations. Estimates may be used. However, final results will be based on actual measurements made during the test.

Select a nozzle size based on the range of velocity heads such that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle size. Ensure that the differential pressure gauge is capable of measuring the minimum velocity head value to within 10 percent, or as specified by the Administrator.

Select a suitable probe liner and probe length such that all traverse points can be sampled. Consider sampling from opposite sides for large stacks to reduce the length of probes.

Select a total sampling time greater than or equal to the minimum total sampling time specified in the test procedures for the specific industry such that the sampling time per point is not less than 2 min. or select some greater time interval as specified by the Administrator, and such that the sample volume that will be taken will exceed the required minimum total gas sample volume specified in the test procedures for the specific industry. The latter is based on an approximate average sampling rate. Note also that the minimum total sample volume is corrected to standard conditions.

It is recommended that a half-integral or integral number of minutes be sampled at each point in order to avoid timekeeping errors.

In some circumstances, e.g. batch cycles, it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes. In these cases, the Administrator's approval must first be obtained.

7.1.3 Preparation of collection train. During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to begin.

Place 100 ml of water in each of the first two impingers, leave the third impinger empty, and place approximately 200–300 g or more, if necessary, of preweighed silica gel in the fourth impinger. Record the weight of the silica gel and container on the data sheet. Place the empty container in a clean place for later use in the sample recovery.

Place a filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as to not allow the sample gas stream to circumvent the filter. Check filter for tears after assembly is completed.

When glass liners are used, install selected nozzle using a Viton A O-ring; the Viton A O-ring is installed as a seal where the nozzle is connected to a glass liner. See APTD-0576 for details. When metal liners are used, install the nozzle as above or by a leak free direct mechanical connection. Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

Unless otherwise specified by the Administrator, attach a temperature probe to the metal sheath of the sampling probe so that the sensor extends beyond the probe tip and does not touch any metal. Its position should be about 1.9 to 2.54 cm (0.75 to 1 in.) from the pitot tube and probe nozzle to avoid interference with the gas flow.

Assemble the train as shown in Figure 13A-1 (Method 13A) with the filter between the third and fourth impingers. Alternatively, the filter may be placed between the probe and first impinger. A filter heating system may be used to prevent moisture condensation, but the temperature around the filter holder shall not exceed 120° ± 14° C (248 ± 25° F). [(Note: Whatman No. 1 filter decomposes at 150° C (300° F)).] Record filter location on the data sheet.

Place crushed ice around the impingers.

7.1.4 Leak check procedure—After the sampling train has been assembled, turn on and set (if applicable) the probe and filter heating system(s) to reach a temperature sufficient to avoid condensation in the probe. Allow time for the temperature to stabilize. Leak check the train at the sampling site by plugging the nozzle and pulling a 380 mm Hg (15 in. Hg) vacuum. A leakage rate in excess of 4% of the average sampling rate of 0.0057 m³/min. (0.02 cfm), whichever is less, is unacceptable.

The following leak check instruction for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with by-pass valve fully open and coarse adjust valve completely closed. Partially open the coarse adjust valve and slowly close the by-pass valve until 380 mm Hg (15 in. Hg) vacuum is reached. *Do not* reverse direction of by-pass valve. This will cause water to back up into the filter holder. If 380 mm Hg (15 in. Hg) is exceeded, either leak check at this higher vacuum or end the leak check as described below and start over.

When the leak check is completed, first slowly remove the plug from the inlet to the probe or filter holder and immediately turn off the vacuum pump. This prevents the water in the impingers from being forced backward into the filter holder (if placed before the impingers) and silica gel from being entrained backward into the third impinger.

Leak checks shall be conducted as described whenever the train is disengaged, e.g. for silica gel or filter changes during the test, prior to each test run, and at the completion of each test run. If leaks are found to be in excess of the acceptable rate, the test will be considered invalid. To reduce lost time due to leakage occurrences, it is recommended that leak checks be conducted between port changes.

7.1.5 Particulate train operation—During the sampling run, an isokinetic sampling rate within 10%, or as specified by the Administrator, of true isokinetic shall be maintained.

For each run, record the data required on the example data sheet shown in Figure 13A-3 (Method 13A). Be sure to record the initial dry gas meter reading. Record the dry gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made, and when sampling is halted. Take other data point readings at least once at each sample point during each time increment and additional readings when significant changes (20% variation in velocity head readings) necessitate additional adjustments in flow rate. Be sure to level and zero the manometer.

Clean the portholes prior to the test run to minimize chance of sampling deposited material. To begin sampling, remove the nozzle cap, verify (if applicable) that the probe heater is working and filter heater is up to temperature, and that the pitot tube and probe are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs are available for sampling trains using type S pitot tubes with 0.85 ± 0.02 coefficients ($C_p$), and when sampling in air or a stack gas with equivalent density (molecular weight, $M_d$, equal to 29±4), which aid in the rapid adjustment of the isokinetic sampling rate without excessive computations. APTD-0576 details the procedure for using these nomographs. If $C_p$ and $M_d$ are outside the above stated ranges, do not use the nomograph unless appropriate steps are taken to compensate for the deviations.

When the stack is under significant negative pressure (height of impinger stem), take care to close the coarse adjust valve before inserting the probe into the stack to avoid water backing into the filter holder. If necessary, the pump may be turned on with the coarse adjust valve closed.

When the probe is in position, block off the openings around the probe and porthole to prevent unrepresentative dilution of the gas stream.

Traverse the stack cross section, as required by Method 1 or as specified by the Administrator, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the portholes to minimize chance of extracting deposited material.

During the test run, make periodic adjustments to keep the probe and (if applicable) filter temperatures at their proper values. Add more ice and, if necessary, salt to the ice bath, to maintain a temperature of less than 20°C (68°F) at the impinger/silica gel outlet, to avoid excessive moisture losses. Also, periodically check the level and zero of the manometer.

If the pressure drop across the filter becomes high enough to make isokinetic sampling difficult to maintain, the filter may be replaced in the midst of a sample run. It is recommended that another complete filter assembly be used rather than attempting to change the filter itself. After the new filter or filter assembly is installed, conduct a leak check. The final emission results shall be based on the summation of all filter catches.

A single train shall be used for the entire sample run, except for filter and silica gel changes. However, if approved by the Administrator, two or more trains may be used for a single test run when there are two or more ducts or sampling ports. The final emission results shall be based on the total of all sampling train catches.

At the end of the sample run, turn off the pump, remove the probe and nozzle from the stack, and record the final dry gas meter reading. Perform a leak check.[1] Calculate percent isokinetic (see calculation section) to determine whether another test run should be made. If there is difficulty in maintaining isokinetic rates due to source conditions, consult with the Administrator for possible variance on the isokinetic rates.

7.2 Sample recovery. Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period.

When the probe can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it to keep from losing part of the sample. Do not cap off the probe tip tightly while the sampling train is cooling down, as this would create a vacuum in the filter holder, thus drawing water from the impingers into the filter.

Before moving the sample train to the cleanup site, remove the probe from the sample train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate, if present. Wipe off the silicone grease from the filter inlet where the probe was fastened and cap it. Remove the umbilical cord from the last impinger and cap the impinger. After wiping off the silicone grease, cap off the filter holder outlet and impinger inlet. Ground glass stoppers, plastic caps, or serum caps may be used to close these openings.

Transfer the probe and filter-impinger assembly to the cleanup area. This area should be clean and protected from the wind so that the chances of contaminating or losing the sample will be minimized.

Inspect the train prior to and during disassembly and note any abnormal conditions. Using a graduated cylinder, measure and record the volume of the water in the first three impingers, to the nearest ml; any condensate in the probe should be included in this determination. Treat the samples as follows:

7.2.1 Container No. 1. Transfer the impinger water from the graduated cylinder to this container. Add the filter to this container. Wash all sample exposed surfaces, including the probe tip, probe, first three impingers, impinger connectors, filter holder, and graduated cylinder thoroughly with distilled water. Wash each component three separate times with water and clean the probe and nozzle with brushes. A maximum wash of 500 ml is used, and the washings are added to the sample container which must be made of polyethylene.

7.2.2 Container No. 2. Transfer the silica gel from the fourth impinger to this container and seal.

7.3 Analysis. Treat the contents of each sample container as described below.

7.3.1 Container No. 1.

7.3.1.1 Filter this container's contents, including the Whatman No 1 filter, through Whatman No. 541 filter paper, or equivalent into a 1500 ml beaker. NOTE: If filtrate volume exceeds 900 ml make filtrate basic with NaOH to phenolphthalein and evaporate to less than 900 ml.

7.3.1.2 Place the Whatman No. 541 filter containing the insoluble matter (including the Whatman No. 1 filter) in a nickel crucible, add a few ml of water and macerate the filter with a glass rod.

Add 100 mg CaO to the crucible and mix the contents thoroughly to form a slurry. Add a couple of drops of phenolphthalein indicator. The indicator will turn red in a basic medium. The slurry should remain basic during the evaporation of the water or fluoride ion will be lost. If the indicator turns colorless during the evaporation, an acidic condition is indicated. If this happens add CaO until the color turns red again.

Place the crucible in a hood under infrared lamps or on a hot plate at low heat. Evaporate the water completely.

After evaporation of the water, place the crucible on a hot plate under a hood and slowly increase the temperature until the paper chars. It may take several hours for complete charring of the filter to occur.

Place the crucible in a cold muffle furnace and gradually (to prevent smoking) increase the temperature to 600°C, and maintain until the contents are reduced to an ash. Remove the crucible from the furnace and allow it to cool.

7.3.1.3 Add approximately 4 g of crushed NaOH to the crucible and mix. Return the crucible to the muffle furnace, and fuse the sample for 10 minutes at 600°C.

Remove the sample from the furnace and cool to ambient temperature. Using several rinsings of warm distilled water transfer the contents of the crucible to the beaker containing the filtrate from container No. 1 (7.3.1). To assure complete sample removal, rinse finally with two 20 ml portions of 25 percent (v/v) sulfuric acid and carefully add to the beaker. Mix well and transfer to a one-liter volumetric flask. Dilute to volume with distilled water and mix thoroughly. Allow any undissolved solids to settle

---

[1] With acceptability of the test run to be based on the same criterion as in 7.1.4.

7.3.2 Container No. 2. Weigh the spent silica gel and report to the nearest 0.5 g.

7.3.3 Adjustment of acid/water ratio in distillation flask—(Utilize a protective shield when carrying out this procedure). Place 400 ml of distilled water in the distilling flask and add 200 ml of concentrated $H_2SO_4$. Caution: Observe standard precautions when mixing the $H_2SO_4$ by slowly adding the acid to the flask with constant swirling. Add some soft glass beads and several small pieces of broken glass tubing and assemble the apparatus as shown in Figure 13A-2. Heat the flask until it reaches a temperature of 175°C to adjust the acid/water ratio for subsequent distillations. Discard the distillate.

7.3.4 Distillation—Cool the contents of the distillation flask to below 80°C. Pipette an aliquot of sample containing less than 0.6 mg F directly into the distilling flask and add distilled water to make a total volume of 220 ml added to the distilling flask. [For an estimate of what size aliquot does not exceed 0.6 mg F, select an aliquot of the solution and treat as described in Section 7.3.6. This will give an approximation of the fluoride content, but only an approximation since interfering ions have not been removed by the distillation step.]

Place a 250 ml volumetric flask at the condenser exit. Now begin distillation and gradually increase the heat and collect all the distillate up to 175°C. Caution: Heating the solution above 175°C will cause sulfuric acid to distill over.

The acid in the distilling flask can be used until there is carryover of interferences or poor fluoride recovery. An occasional check of fluoride recovery with standard solutions is advised. The acid should be changed whenever there is less than 90 percent recovery or blank values are higher than 0.1 μg/ml.

7.3.5 Determination of concentration—Bring the distillate in the 250 ml volumetric flask to the mark with distilled water and mix thoroughly. Pipette a 25 ml aliquot from the distillate. Add an equal volume of TISAB and mix. The sample should be at the same temperature as the calibration standards when measurements are made. If ambient lab temperature fluctuates more than ±2°C from the temperature at which the calibration standards were measured, condition samples and standards in a constant temperature bath measurement. Stir the sample with a magnetic stirrer during measurement to minimize electrode response time. If the stirrer generates enough heat to change solution temperature, place a piece of insulating material such as cork between the stirrer and the beaker. Dilute samples (below $10^{-4}$ M fluoride ion content) should be held in polyethylene or polypropylene beakers during measurement.

Insert the fluoride and reference electrodes into the solution. When a steady millivolt reading is obtained, record it. This may take several minutes. Determine concentration from the calibration curve. Between electrode measurements, soak the fluoride sensing electrode in distilled water for 30 seconds and then remove and blot dry.

8. *Calibration.*

Maintain a laboratory log of all calibrations.

8.1 Sampling Train.

8.1.1 Probe nozzle—Using a micrometer, measure the inside diameter of the nozzle to the nearest 0.025 mm (0.001 in.). Make 3 separate measurements using different diameters each time and obtain the average of the measurements. The difference between the high and low numbers shall not exceed 0.1 mm (0.004 in.).

When nozzles become nicked, dented, or corroded, they shall be reshaped, sharpened, and recalibrated before use.

Each nozzle shall be permanently and uniquely identified.

8.1.2 Pitot tube—The pitot tube shall be calibrated according to the procedure outlined in Method 2.

8.1.3 Dry gas meter and orifice meter. Both meters shall be calibrated according to the procedure outlined in APTD-0576. When diaphragm pumps with by-pass valves are used, check for proper metering system design by calibrating the dry gas meter at an additional flow rate of 0.0057 m³/min. (0.2 cfm) with the by-pass valve fully opened and then with it fully closed. If there is more than ±2 percent difference in flow rates when compared to the fully closed position of the by-pass valve, the system is not designed properly and must be corrected.

8.1.4 Probe heater calibration—The probe heating system shall be calibrated according to the procedure contained in APTD-0576. Probes constructed according to APTD-0581 need not be calibrated if the calibration curves in APTD-0576 are used.

8.1.5 Temperature gauges—Calibrate dial and liquid filled bulb thermometers against mercury-in-glass thermometers. Thermocouples need not be calibrated. For other devices, check with the Administrator.

8.2 Analytical Apparatus.

8.2.1 Fluoride Electrode—Prepare fluoride standardizing solutions by serial dilution of the 0.1 M fluoride standard solution. Pipette 10 ml of 0.1 M NaF into a 100 ml volumetric flask and make up to the mark with distilled water for a $10^{-2}$ M standard solution. Use 10 ml of $10^{-2}$ M solution to make a $10^{-3}$ M solution in the same manner. Repeat for $10^{-4}$ and $10^{-5}$ M solutions.

Pipette 50 ml of each standard into a separate beaker. Add 50 ml of TISAB to each beaker. Place the electrode in the most dilute standard solution. When a steady millivolt reading is obtained, plot the value on the linear axis of semi-log graph paper versus concentration on the log axis. Plot the nominal value for concentration of the standard on the log axis, e.g., when 50 ml of $10^{-2}$ M standard is diluted with 50 ml TISAB, the concentration is still designated "$10^{-2}$ M".

Between measurements soak the fluoride sensing electrode in distilled water for 30 seconds, and then remove and blot dry. Analyze the standards going from dilute to concentrated standards. A straight-line calibration curve will be obtained, with nominal concentrations of $10^{-5}$, $10^{-4}$, $10^{-3}$, $10^{-2}$, $10^{-1}$ fluoride molarity on the log axis plotted versus electrode potential (in millivolts) on the linear scale.

Calibrate the fluoride electrode daily, and check it hourly. Prepare fresh fluoride standardizing solutions daily of $10^{-2}$ M or less. Store fluoride standardizing solutions in polyethylene or polypropylene containers. (Note: Certain specific ion meters have been designed specifically for fluoride electrode use and give a direct readout of fluoride ion concentration. These meters may be used in lieu of calibration curves for fluoride measurements over narrow concentration ranges. Calibrate the meter according to manufacturer's instructions.)

9. *Calculations.*

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

9.1 Nomenclature.

$A_n$ = Cross sectional area of nozzle, m² (ft²).

$A_t$ = Aliquot of total sample added to still, ml.

$B_{ws}$ = Water vapor in the gas stream, proportion by volume.

$C_s$ = Concentration of fluoride in stack gas, mg/m³, corrected to standard conditions of 20° C, 760 mm Hg (68° F, 29.92 in. Hg) on dry basis.

$F_t$ = Total weight of fluoride in sample, mg.

$I$ = Percent of isokinetic sampling.

$M$ = Concentration of fluoride from calibration curve, molarity.

$m_n$ = Total amount of particulate matter collected, mg.

$M_w$ = Molecular weight of water, 18 g/g-mole (18 lb/lb-mole).

$m_a$ = Mass of residue of acetone after evaporation, mg.

$P_{bar}$ = Barometric pressure at the sampling site, mm Hg (in. Hg).

$P_s$ = Absolute stack gas pressure, mm Hg (in. Hg).

$P_{std}$ = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

$R$ = Ideal gas constant, 0.06236 mm Hg-m³/°K-g-mole (21.83 in. Hg-ft³/°R-lb-mole).

$T_m$ = Absolute average dry gas meter temperature (see fig. 13A-3), °K (°R).

$T_s$ = Absolute average stack gas temperature (see fig. 13A-3), °K (°R).

$T_{std}$ = Standard absolute temperature, 293° K (528° R).

$V_a$ = Volume of acetone blank, ml.

$V_{aw}$ = Volume of acetone used in wash, ml.

$V_d$ = Volume of distillate collected, ml.

$V_{lc}$ = Total volume of liquid collected in impingers and silica gel, ml. Volume of water in silica gel equals silica gel weight increase in grams times 1 ml/gram. Volume of liquid collected in impinger equals final volume minus initial volume.

$V_m$ = Volume of gas sample as measured by dry gas meter, dcm (dcf).

$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter corrected to standard conditions, dscm (dscf).

$V_{w(std)}$ = Volume of water vapor in the gas sample corrected to standard conditions, scm (scf).

$V_t$ = Total volume of sample, ml.

$v_s$ = Stack gas velocity, calculated by Method 2, Equation 2-7 using data obtained from Method 5, m/sec (ft/sec).

$W_a$ = Weight of residue in acetone wash, mg.

$\Delta H$ = Average pressure differential across the orifice (see fig. 13A-3), meter, mm $H_2O$ (in. $H_2O$).

$\rho_a$ = Density of acetone, mg/ml (see label on bottle).

$\rho_w$ = Density of water, 1 g/ml (0.00220 lb/ml).

$\theta$ = Total sampling time, min.

13.6 = Specific gravity of mercury.

60 = Sec/min.

100 = Conversion to percent.

9.2 Average dry gas meter temperature and average orifice pressure drop. See data sheet (Figure 13A-3 of Method 13A).

9.3 Dry gas volume. Use Section 9.3 of Method 13A.

9.4 Volume of Water Vapor. Use Section 9.4 of Method 13A.

9.5 Moisture Content. Use Section 9.5 of Method 13A.

9.6 Concentration

9.6.1 Calculate the amount of fluoride in the sample according to equation 13B-1.

$$F_t = K \frac{V_t}{A_t} (V_d)\ (M)$$

where:

$K$ = 19 mg/ml.

9.6.2 Concentration of fluoride in stack gas. Use Section 9.6.2 of Method 13A.

9.7 Isokinetic variation. Use Section 9.7 of Method 13A.

9.8 Acceptable results. Use Section 9.8 of Method 13A.

10. *References.*

Bellack, Ervin, "Simplified Fluoride Distillation Method," *Journal of the American Water Works Association* #50: 530-6 (1958).

MacLeod, Kathryn E., and Howard L. Crist, "Comparison of the SPADNS—Zirconium Lake and Specific Ion Electrode Methods of Fluoride Determination in Stack Emission Samples," *Analytical Chemistry* 45: 1272-1273 (1973).

Martin, Robert M. "Construction Details of Isokinetic Source Sampling Equipment," Environmental Protection Agency, Air Pollution Control Office Publication No. APTD-0581.

*1973 Annual Book of ASTM Standards*, Part 23, Designation: D 1179-72.

Pom, Jerome J., "Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment," Environmental Protection Agency, Air Pollution Control Office Publication No. APTD-0576.

*Standard Methods for the Examination of Water and Waste Water*, published jointly by American Public Health Association, American Water Works Association and Water Pollution Control Federation, 13th Edition (1971).

Reference Method 13B is amended as follows:

(a) In the third line of section 3, the phrase "300μg/liter" is corrected to read "300 mg/liter".

(b) Section 5.1.5 is revised to read as follows:

> 5.1.5 Filter holder—If located between the probe and first impinger, borosilicate glass with a 20 mesh stainless steel screen filter support and a silicone rubber gasket; neither a glass frit filter support nor a sintered metal filter support may be used if the filter is in front of the impingers. If located between the third and fourth impingers, borosilicate glass with a glass frit filter support and a silicone rubber gasket. Other materials of construction may be used with approval from the Administrator, e.g., if probe liner is stainless steel, then filter holder may be stainless steel. The holder design shall provide a positive seal against leakage from the outside or around the filter.

(c) Section 7.1.3 is amended by revising the first two sentences of the sixth paragraph to read as follows:

> 7.1.3 Preparation of collection train. * * * Assemble the train as shown in Figure 13A-1 (Method 13A) with the filter between the third and fourth impingers. Alternatively, the filter may be placed between the probe the first impinger if a 20 mesh stainless steel screen is used for the filter support. * * *

* * * * *

(d) In section 7.3.4, the reference in the first paragraph to "section 7.3.6" is corrected to read "section 7.3.5".

# METHOD 14

# DETERMINATION OF FLUORIDE EMISSIONS FROM POT ROOM ROOF MONITORS OF PRIMARY ALUMINUM PLANTS

1. *Principle and applicability.*

1.1 *Principle.* Gaseous and particulate fluoride roof monitor emissions are drawn into a permanent sampling manifold through several large nozzles. The sample is transported from the sampling manifold to ground level through a duct. The gas in the duct is sampled using Method 13A or 13B—DETERMINATION OF TOTAL FLUORIDE EMISSIONS FROM STATIONARY SOURCES. Effluent velocity and volumetric flow rate are determined with anemometers permanently located in the roof monitor.

1.2 *Applicability.* This method is applicable for the determination of fluoride emissions from stationary sources only when specified by the test procedures for determining compliance with new source performance standards.

2. *Apparatus.*

2.1 *Velocity measurement apparatus.*

2.1.1 *Anemometers.* Vane or propeller anemometers with a velocity measuring threshold as low as 15 meters/minute and a range up to at least 600 meters/minute. Each anemometer shall generate an electrical signal which can be calibrated to the velocity measured by the anemometer. Anemometers shall be able to withstand dusty and corrosive atmospheres.

One anemometer shall be installed for every 85 meters of roof monitor length. If the roof monitor length divided by 85 meters is not a whole number, round the fraction to the nearest whole number to determine the number of anemometers needed. Use one anemometer for any roof monitor less than 85 meters long. Permanently mount the anemometers at the center of each equal length along the roof monitor. One anemometer shall be installed in the same section of the roof monitor that contains the sampling manifold (see section 2.2.1). Make a velocity traverse of the width of the roof monitor where an anemometer is to be placed. This traverse may be made with any suitable low velocity measuring device, and shall be made during normal process operating conditions. Install the anemometer at a point of average velocity along this traverse.

2.1.2 *Recorders.* Recorders equipped with signal transducers for converting the electrical signal from each anemometer to a continuous recording of air flow velocity, or to an integrated measure of volumetric flow. For the purpose of recording velocity, "continuous" shall mean one readout per 15-minute or shorter time interval. A constant amount of time shall elapse between readings. Volumetric flow rate may be determined by an electrical count of anemometer revolutions. The recorders or counters shall permit identification of the velocities or flow rate measured by each individual anemometer.

2.2 Roof monitor air sampling system.

2.2.1 *Sampling ductwork.* The manifold system and connecting duct shall be permanently installed to draw an air sample from the roof monitor to ground level. A typical installation of duct for drawing a sample from a roof monitor to ground level is shown in Figure 14-1. A plan of a manifold system that is located in a roof monitor is shown in Figure 14-2. These drawings represent a typical installation for a generalized roof monitor. The dimensions on these figures may be altered slightly to make the manifold system fit into a particular roof monitor, but the general configuration shall be followed. There shall be eight nozzles, each having a diameter of 0.40 to 0.50 meters. The length of the manifold system from the first nozzle to the eighth shall be 35 meters or eight percent of the length of the roof monitor, whichever is greater. The duct leading from the roof monitor manifold shall be round with a diameter of 0.30 to 0.40 meters. As shown in Figure 14-2, each of the sample legs of the manifold shall have a device, such as a blast gate or valve, to enable adjustment of flow into each sample nozzle.

Locate the manifold along the length of the roof monitor so that it lies near the midsection of the roof monitor. If the design of a particular roof monitor makes this impossible, the manifold may be located elsewhere along the roof monitor, but avoid locating the manifold near the ends of the roof monitor or in a section where the aluminum reduction pot arrangement is not typical of the rest of the potroom. Center the sample nozzles in the throat of the roof monitor. (See Figure 14-1.) Construct all

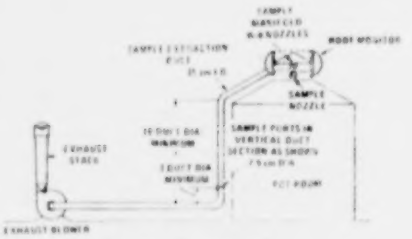


Figure 14-1. Roof Monitor Sampling System.

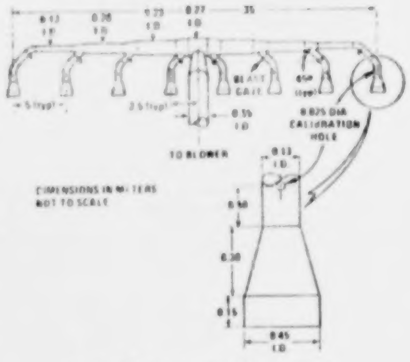


Figure 14-2. Sampling Manifold and Nozzles.

Reproduced from best available copy.

sample-exposed surfaces within the nozzles, manifold and sample duct of 316 stainless steel. Aluminum may be used if a new ductwork system is conditioned with fluoride-laden roof monitor air for a period of six weeks prior to initial testing. Other materials of construction may be used if it is demonstrated through comparative testing that there is no loss of fluorides in the system. All connections in the ductwork shall be leak free.

Locate two sample ports in a vertical section of the duct between the roof monitor and exhaust fan. The sample ports shall be at least 10 duct diameters downstream and two diameters upstream from any flow disturbance such as a bend or contraction. The two sample ports shall be situated 90° apart. One of the sample ports shall be situated so that the duct can be traversed in the plane of the nearest upstream duct bend.

2.2.2 *Exhaust fan*. An industrial fan or blower to be attached to the sample duct at ground level. (See Figure 14-1.) This exhaust fan shall have a maximum capacity such that a large enough volume of air can be pulled through the ductwork to maintain an isokinetic sampling rate in all the sample nozzles for all flow rates normally encountered in the roof monitor.

The exhaust fan volumetric flow rate shall be adjustable so that the roof monitor air can be drawn isokinetically into the sample nozzles. This control of flow may be achieved by a damper on the inlet to the exhauster or by any other workable method.

2.3 Temperature measurement apparatus.

2.3.1 *Thermocouple*. Installed in the roof monitor near the sample duct.

2.3.2 *Signal transducer*. Transducer to change the thermocouple voltage output to a temperature readout.

2.3.3 *Thermocouple wire*. To reach from roof monitor to signal transducer and recorder.

2.3.4 *Sampling train*. Use the train described in Methods 13A and 13B—Determination of total fluoride emissions from stationary sources.

3. *Reagents*.

3.1 *Sampling and analysis*. Use reagents described in Method 13A or 13B—Determination of total fluoride emissions from stationary sources.

4. *Calibration*.

4.1 *Propeller anemometer*. Calibrate the anemometers so that their electrical signal output corresponds to the velocity or volumetric flow they are measuring. Calibrate according to manufacturer's instructions.

4.2 *Manifold intake nozzles*. Adjust the exhaust fan to draw a volumetric flow rate (refer to Equation 14-1) such that the entrance velocity into each manifold nozzle approximates the average effluent velocity in the roof monitor. Measure the velocity of the air entering each nozzle by inserting an S type pitot tube into a 2.5 cm or less diameter hole (see Figure 14-2) located in the manifold between each blast gate (or valve) and nozzle. The pitot tube tip shall be extended into the center of the manifold. Take care to insure that there is no leakage around the pitot probe which could affect the indicated velocity in the manifold leg. If the velocity of air being drawn into each nozzle is not the same, open or close each blast gate (or valve) until the velocity in each nozzle is the same. Fasten each blast gate (or valve) so that it will remain in this position and close the pitot port holes. This calibration shall be performed when the manifold system is installed. (Note: It is recommended that this calibration be repeated at least once a year.)

5. *Procedure*.

5.1 *Roof monitor velocity determination*.

5.1.1 *Velocity value for setting isokinetic flow*. During the 24 hours preceding a test run, determine the velocity indicated by the propeller anemometer in the section of roof monitor containing the sampling manifold. Velocity readings shall be taken every 15 minutes or at shorter equal time intervals. Calculate the average velocity for the 24-hour period.

5.1.2 *Velocity determination during a test run*. During the actual test run, record the velocity or volume readings of each propeller anemometer in the roof monitor. Velocity readings shall be taken for each anemometer every 15 minutes or at shorter equal time intervals (or continuously).

5.2 *Temperature recording*. Record the temperature of the roof monitor every two hours during the test run.

5.3 *Sampling*.

5.3.1 *Preliminary air flow in duct*. During the 24 hours preceding the test, turn on the exhaust fan and draw roof monitor air through the manifold duct to condition the ductwork. Adjust the fan to draw a volumetric flow through the duct such that the velocity of gas entering the manifold nozzles approximates the average velocity of the air leaving the roof monitor.

5.3.2 *Isokinetic sample rate adjustment*. Adjust the fan so that the volumetric flow rate in the duct is such that air enters into the manifold sample nozzles at a velocity equal to the 24-hour average velocity determined under 5.1.1. Equation 14-1 gives the correct stream velocity which is needed in the duct at the sample ports in order for sample gas to be drawn isokinetically into the manifold nozzles. Perform a pitot traverse of the duct at the sample ports to determine if the correct average velocity in the duct has been achieved. Perform the pitot determination according to Method 2. Make this determination before the start of a test run. The fan setting need not be changed during the run.

$$V_d = \frac{8\,(D_n)^2}{(D_d)^2}\,(V_m)\,\frac{1\text{ minute}}{60\text{ sec}} \qquad \text{Eq. 14-1}$$

where:

$V_d$=desired velocity in duct at sample ports, meter/sec.

$D_n$=diameter of a roof monitor manifold nozzle, meters.

$D_d$=diameter of duct at sample port, meters.

$V_m$=average velocity of the air stream in the roof monitor, meters/minute, as determined under section 5.1.1.

5.3.3 *Sample train operation*. Sample the duct using the standard fluoride train and methods described in Methods 13A and 13B—Determination of total fluoride emissions from stationary sources. Select sample traverse points according to Method 1. If a selected sampling point is less than one inch from the stack wall, adjust the location of that point to one inch away from the wall.

5.3.4 *Each test run shall last eight hours or more*. If a question exists concerning the representativeness of an eight-hour test, a longer test period up to 24 hours may be selected. Conduct each run during a period when all normal operations are performed underneath the sampling manifold, i.e. tapping, anode changes, maintenance, and other normal duties. All pots in the potroom shall be operated in a normal manner during the test period.

5.3.5 *Sample recovery*. Same as Method 13A or 13B—Determination of total fluoride emissions from stationary sources.

5.4 *Analysis*. Same as Method 13A or 13B—Determination of total fluoride emissions from stationary sources.

6. *Calculations*.

6.1 *Isokinetic sampling test*. Calculate the mean velocity measured during each sampling run by the anemometer in the section of the roof monitor containing the sampling manifold. If the mean velocity recorded during a particular test run does not fall within ±20 percent of the mean velocity established according to 5.3.2, repeat the run.

6.2 *Average velocity of roof monitor gases* Calculate the average roof monitor velocity using all the velocity or volumetric flow readings from section 5.1.2.

6.3 *Roof monitor temperature*. Calculate the mean value of the temperatures recorded in section 5.2.

6.4 *Concentration of fluorides in roof monitor air in mg F/m³*. This is given by Equation 13A-5 in Method 13A—Determination of total fluoride emissions from stationary sources.

6.5 Average volumetric flow from roof is given by Equation 14-2.

$$Q_m = \frac{V_{mt}\,(A)\,(M_d)\,P_m\,(294\,^{\circ}\mathrm{K})}{(T_m + 273^{\circ})\,(760\ \mathrm{mm\ Hg})} \qquad \text{Eq. 14-2}$$

where:

$Q_m$=average volumetric flow from roof monitor at standard conditions on a dry basis, m³/min.

$A$=roof monitor open area, m².

$V_{mt}$=average velocity of air in the roof monitor, meters/minute, from section 6.2.

$P_m$=atmospheric pressure, mm Hg.

$T_m$=roof monitor temperature, °C, from section 6.3.

$M_d$=mole fraction of dry gas, which is given by $M_d = \frac{100 - 100\,(B_{wo})}{100}$

$B_{wo}$=is the proportion by volume of water vapor in the gas stream, from Equation 13A-3, Method 13A—Determination of total fluoride emissions from stationary sources.

[36 FR 24877, Dec. 23, 1971, as amended at 38 FR 13562, May 23, 1973; 39 FR 9319, Mar. 8, 1974; 39 FR 13776, Apr. 17, 1974; 39 FR 20794, June 14, 1974; 39 FR 39874, Nov. 12, 1974; 40 FR 33157, Aug. 6, 1975; 41 FR 3828, Jan. 26, 1976]

# METHOD 15

# DETERMINATION OF $H_2S$, COS, and $CS_2$ EMISSIONS FROM STATIONARY SOURCES

## INTRODUCTION

The method described below uses the principle of gas chromatographic separation and flame photometric detection (FPD). Since there are many systems or sets of operating conditions that represent usable methods of determining sulfur emissions, all systems which employ this principle, but differ only in details of equipment and operation, may be used as alternative methods, provided that the criteria set below are met.

### *1. Principle and applicability*

1.1 Principle. A gas sample is extracted from the emission source and diluted with clean dry air. An aliquot of the diluted sample is then analyzed for hydrogen sulfide ($H_2S$), carbonyl sulfide (COS), and carbon disulfide ($CS_2$) by gas chromatographic (GC) separation and flame photometric detection (FPD).

1.2 Applicability. This method is applicable for determination of the above sulfur compounds from tail gas control units of sulfur recovery plants.

### *2. Range and sensitivity*

2.1 Range. Coupled with a gas chromatographic system utilizing a 1-milliliter sample size, the maximum limit of the FPD for each sulfur compound is approximately 10 ppm. It may be necessary to dilute gas samples from sulfur recovery plants hundredfold (99:1) resulting in an upper limit of about 1000 ppm for each compound.

2.2 The minimum detectable concentration of the FPD is also dependent on sample size and would be about 0.5 ppm for a 1 ml sample.

### *3. Interferences*

3.1 Moisture Condensation. Moisture condensation in the sample delivery system, the analytical column, or the FPD burner block can cause losses or interferences. This potential is eliminated by heating the sample line, and by conditioning the sample with dry dilution air to lower its dew point below the operating temperature of the GC/FPD analytical system prior to analysis.

3.2 Carbon Monoxide and Carbon Dioxide. CO and $CO_2$ have substantial desensitizing effects on the flame photometric detector even after 9:1 dilution. (Acceptable systems must demonstrate that they have eliminated this interference by some procedure such as eluting CO and $CO_2$ before any of the sulfur compounds to be measured.) Compliance with this requirement can be demonstrated by submitting chromatograms of calibration gases with and without $CO_2$ in the diluent gas. The $CO_2$ level should be approximately 10 percent for the case with $CO_2$ present. The two chromatographs should show agreement within the precision limits of section 4.1.

3.3 Elemental Sulfur. The condensation of sulfur vapor in the sampling line can lead to eventual coating and even blockage of the sample line. This problem can be eliminated along with the moisture problem by heating the sample line.

### *4. Precision*

4.1 Calibration Precision. A series of three consecutive injections of the same calibration gas, at any dilution, shall produce results which do not vary by more than ±13 percent from the mean of the three injections.

4.2 Calibration Drift. The calibration drift determined from the mean of three injections made at the beginning and end of any 8-hour period shall not exceed ±5 percent.

### *5. Apparatus*

5.1.1 Probe. The probe must be made of inert material such as stainless steel or glass. It should be designed to incorporate a filter and to allow calibration gas to enter the probe at or near the sample entry point. Any portion of the probe not exposed to the stack gas must be heated to prevent moisture condensation.

5.1.2 The sample line must be made of Teflon,[1] no greater than 1.3 cm (½ in) inside diameter. All parts from the probe to the dilution system must be thermostatically heated to 120° C.

5.1.3 Sample Pump. The sample pump shall be a leakless Teflon coated diaphragm type or equivalent. If the pump is upstream of the dilution system, the pump head must be heated to 120° C.

5.2 Dilution System. The dilution system must be constructed such that all sample contacts are made of inert material (e.g. stainless steel or Teflon). It must be heated to 120° C and be capable of approximately a 9:1 dilution of the sample.

5.3 Gas Chromatograph. The gas chromatograph must have at least the following components:

5.3.1 Oven. Capable of maintaining the separation column at the proper operating temperature ±1° C.

5.3.2 Temperature Gauge. To monitor column oven, detector, and exhaust temperature ±1° C.

5.3.3 Flow System. Gas metering system to measure sample, fuel, combustion gas, and carrier gas flows.

---

[1] Mention of trade names or specific products does not constitute an endorsement by the Environmental Protection Agency.

Reproduced from
best available copy.

5.3.4 Flame Photometric Detector.

5.3.4.1 Electrometer. Capable of full scale amplification of linear ranges of $10^{-9}$ to $10^{-4}$ amperes full scale.

5.3.4.2 Power Supply. Capable of delivering up to 750 volts.

5.3.4.3 Recorder. Compatible with the output voltage range of the electrometer.

**5.4 Gas Chromatograph Columns.** The column system must be demonstrated to be capable of resolving three major reduced sulfur compounds: $H_2S$, COS, and $CS_2$.

To demonstrate that adequate resolution has been achieved the tester must submit a chromatograph of a calibration gas containing all three reduced sulfur compounds in the concentration range of the applicable standard. Adequate resolution will be defined as base line separation of adjacent peaks when the amplifier attenuation is set so that the smaller peak is at least 50 percent of full scale. Base line separation is defined as a return to zero ±5 percent in the interval between peaks. Systems not meeting this criteria may be considered alternate methods subject to the approval of the Administrator.

5.5.1 Calibration System. The calibration system must contain the following components.

**5.5.2 Flow System.** To measure air flow over permeation tubes at ±2 percent. Each flowmeter shall be calibrated after a complete test series with a wet test meter. If the flow measuring device differs from the wet test meter by 5 percent, the completed test shall be discarded. Alternatively, the tester may elect to use the flow data that would yield the lowest flow measurement. Calibration with a wet test meter before a test is optional.

**5.5.3 Constant Temperature Bath.** Device capable of maintaining the permeation tubes at the calibration temperature within ±1.1° C.

5.5.4 Temperature Gauge. Thermometer or equivalent to monitor bath temperature within ±1° C.

## *6. Reagents*

6.1 Fuel. Hydrogen ($H_2$) prepurified grade or better.

6.2 Combustion Gas. Oxygen ($O_2$) or air, research purity or better.

6.3 Carrier Gas. Prepurified grade or better.

6.4 Diluent. Air containing less than 0.5 ppm total sulfur compounds and less than 10 ppm each of moisture and total hydrocarbons.

6.5 Calibration Gases. Permeation tubes, one each of $H_2S$, COS, and $CS_2$, gravimetrically calibrated and certified at some convenient operating temperature. These tubes consist of hermetically sealed FEP Teflon tubing in which a liquified gaseous substance is enclosed. The enclosed gas permeates through the tubing wall at a constant rate. When the temperature is constant, calibration gases covering a wide range of known concentrations can be generated by varying and accurately measuring the flow rate of diluent gas passing over the tubes. These calibration gases are used to calibrate the GC/FPD system and the dilution system.

## *7. Pretest Procedures*

The following procedures are optional but would be helpful in preventing any problem which might occur later and invalidate the entire test.

7.1 After the complete measurement system has been set up at the site and deemed to be operational, the following procedures should be completed before sampling is initiated.

7.1.1 Leak Test. Appropriate leak test procedures should be employed to verify the integrity of all components, sample lines, and connections. The following leak test procedure is suggested: For components upstream of the sample pump, attach the probe end of the sample line to a manometer or vacuum gauge, start the pump and pull greater than 50 mm (2 in.) Hg vacuum, close off the pump outlet, and then stop the pump and ascertain that there is no leak for 1 minute. For components after the pump, apply a slight positive pressure and check for leaks by applying a liquid (detergent in water, for example) at each joint. Bubbling indicates the presence of a leak.

7.1.2 System Performance. Since the complete system is calibrated following each test, the precise calibration of each component is not critical. However, these components should be verified to be operating properly. This verification can be performed by observing the response of flowmeters or of the GC output to changes in flow rates or calibration gas concentrations and ascertaining the response to be within predicted limits. If any component or the complete system fails to respond in a normal and predictable manner, the source of the discrepancy should be identifed and corrected before proceeding.

## *8. Calibration*

Prior to any sampling run, calibrate the system using the following procedures. (If more than one run is performed during any 24-hour period, a calibration need not be performed prior to the second and any subsequent runs. The calibration must, however, be verified as prescribed in section 10, after the last run made within the 24-hour period.)

8.1 General Considerations. This section outlines steps to be followed for use of the GC/FPD and the dilution system. The procedure does not include detailed instructions because the operation of these systems is complex, and it requires an understanding of the individual system being used. Each system should include a written operating manual describing in detail the operating procedures associated with each component in the measurement system. In addition, the operator shuld be familiar with the operating principles of the components; particularly the GC/FPD. The citations in the Bibliography at the end of this method are recommended for review for this purpose.

8.2 Calibration Procedure. Insert the permeation tubes into the tube chamber. Check the bath temperature to assure agreement with the calibration temperature of the tubes within ±0.1° C. Allow 24 hours for the tubes to equilibrate. Alternatively equilibration may be verified by injecting samples of calibration gas at 1-hour intervals. The permeation tubes can be assumed to have reached equilibrium when consecutive hourly samples agree within the precision limits of section 4.1.

Vary the amount of air flowing over the tubes to produce the desired concentrations for calibrating the analytical and dilution systems. The air flow across the tubes must at all times exceed the flow requirement of the analytical systems. The concentration in parts per million generated by a tube containing a specific permeant can be calculated as follows:

$$C = K \times P_r / ML$$

Equation 15-1

where:

C = Concentration of permeant produced in ppm.

$P_r$ = Permeation rate of the tube in µg/min.

M = Molecular weight of the permeant: g/g-mole.

L = Flow rate, l/min, of air over permeant @ 20°C, 760 mm Hg.

K = Gas constant at 20°C and 760 mm Hg = 24.04 l/g mole.

8.3 Calibration of analysis system. Generate a series of three or more known concentrations spanning the linear range of the FPD (approximately 0.05 to 1.0 ppm) for each of the four major sulfur compounds. Bypassing the dilution system, inject these standards in to the GC/FPD analyzers and monitor the responses. Three injects for each concentration must yield the precision described in section 4.1. Failure to attain this precision is an indication of a problem in the calibration or analytical system. Any such problem must be identified and corrected before proceeding.

8.4 Calibration Curves. Plot the GC/FPD response in current (amperes) versus their causative concentrations in ppm on log-log coordinate graph paper for each sulfur compound. Alternatively, a least squares equation may be generated from the calibration data.

8.5 Calibration of Dilution System. Generate a know concentration of hydrogen sulfied using the permeation tube system. Adjust the flow rate of diluent air for the first dilution stage so that the desired level of dilution is approximated. Inject the diluted calibration gas into the GC/FPD system and monitor its response. Three injections for each dilution must yield the precision described in section 4.1. Failure to attain this precision in this step is an indication of a problem in the dilution system. Any such problem must be identified and corrected before proceeding. Using the calibration data for $H_2S$ (developed under 8.3) determine the diluted calibration gas concentration in ppm. Then calculate the dilution factor as the ratio of the calibration gas concentration before dilution to the diluted calibration gas concentration determined under this paragraph. Repeat this procedure for each stage of dilution required. Alternatively, the GC/FPD system may be calibrated by generating a series of three or more concentrations of each sulfur compound and diluting these samples before injecting them into the GC/FPD system. This data will then serve as the calibration data for the unknown samples and a separate determination of the dilution factor will not be necessary. However, the precision requirements of section 4.1 are still applicable.

### *9. Sampling and Analysis Procedure*

9.1 Sampling. Insert the sampling probe into the test port making certain that no dilution air enters the stack through the port. Begin sampling and dilute the sample approximately 9:1 using the dilution system. Note that the precise dilution factor is that which is determined in paragraph 8.5. Condition the entire system with sample for a minimum of 15 minutes prior to commencing analysis.

9.2 Analysis. Aliquots of diluted sample are injected into the GC/FPD analyzer for analysis.

9.2.1 Sample Run. A sample run is composed of 16 individual analyses (injects) performed over a period of not less than 3 hours or more than 6 hours.

9.2.2 Observation for Clogging of Probe. If reductions in sample concentrations are observed during a sample run that cannot be explained by process conditions, the sampling must be interrupted to determine if the sample probe is clogged with particulate matter. If the probe is found to be clogged, the test must be stopped and the results up to that point discarded. Testing may resume after cleaning the probe or replacing it with a clean one. After each run, the sample probe must be inspected and, if necessary, dismantled and cleaned.

### *10. Post-Test Procedures*

10.1 Sample Line Loss. A known concentration of hydrogen sulfide at the level of the applicable standard, ±20 percent, must be introduced into the sampling system at the opening of the probe in sufficient quantities to ensure that there is an excess of sample which must be vented to the atmosphere. The sample must be transported through the entire sampling system to the measurement system in the normal manner. The resulting measured concentration should be compared to the known value to determine the sampling system loss. A sampling system loss of more than 20 percent is unacceptable. Sampling losses of 0-20 percent must be corrected by dividing the resulting sample concentration by the fraction of recovery. The known gas sample may be generated using permeation tubes. Alternatively, cylinders of hydrogen sulfide mixed in air may be used provided they are traceable to permeation tubes. The optional pretest procedures provide a good guideline for determining if there are leaks in the sampling system.

10.2 Recalibration. After each run, or after a series of runs made within a 24-hour period, perform a partial recalibration using the procedures in section 8. Only $H_2S$ (or other permeant) need be used to recalibrate the GC/FPD analysis system (8.3) and the dilution system (8.5).

10.3 Determination of Calibration Drift. Compare the calibration curves obtained prior to the runs, to the calibration curves obtained under paragraph 10.1. The calibration drift should not exceed the limits set forth in paragraph 4.2. If the drift exceeds this limit, the intervening run or runs should be considered not valid. The tester, however, may instead have the option of choosing the calibration data set which would give the highest sample values.

### *11. Calculations*

11.1 Determine the concentrations of each reduced sulfur compound detected directly from the calibration curves. Alternatively, the concentrations may be calculated using the equation for the least squares line.

11.2 Calculation of $SO_2$ Equivalent. $SO_2$ equivalent will be determined for each analysis made by summing the concentrations of each reduced sulfur compound resolved during the given analysis.

$$SO_2 \text{ equivalent} = \Sigma(H_2S, COS, 2\,CS_2)d$$

Equation 15-2

where:

$SO_2$ equivalent = The sum of the concentration of each of the measured compounds ($COS$, $H_2S$, $CS_2$) expressed as sulfur dioxide in ppm.

$H_2S$ = Hydrogen sulfide, ppm.

$COS$ = Carbonyl sulfide, ppm.

$CS_2$ = Carbon disulfide, ppm.

d = Dilution factor, dimensionless.

11.3 Average $SO_2$ equivalent will be determined as follows:

$$\text{Average } SO_2 \text{ equivalent} = \frac{\sum_{i=1}^{N} SO_2 \text{ equiv.}_i}{N\,(1 - Bwo)}$$

Equation 15-3

where:

Average $SO_2$ equivalent = Average $SO_2$ equivalent in ppm, dry basis.

Average $SO_2$ equivalent$_i$ = $SO_2$ in ppm as determined by Equation 15-2.

N = Number of analyses performed.

Bwo = Fraction of volume of water vapor in the gas stream as determined by Method 4--Determination of Moisture in Stack Gases (36 FR 24887).

### *12. Example System*

Described below is a system utilized by EPA in gathering NSPS data. This system does not now reflect all the latest developments in equipment and column technology, but it does represent one system that has been demonstrated to work.

12.1 Apparatus.

12.1.1 Sample System.

12.1.1.1 Probe. Stainless steel tubing, 6.35 mm (¼ in.) outside diameter, packed with glass wool.

12.1.1.2 Sample Line. 3/16 inch inside diameter Teflon tubing heated to 120° C. This temperature is controlled by a thermostatic heater.

12.1.1.3 Sample Pump. Leakless Teflon coated diaphragm type or equivalent. The pump head is heated to 120° C by enclosing it in the sample dilution box (12.2.4 below).

12.1.2 Dilution System. A schematic diagram of the dynamic dilution system is given in Figure 15-2. The dilution system is constructed such that all sample contacts are made of inert materials. The dilution system which is heated to 120° C must be capable of a minimum of 9:1 dilution of sample. Equipment used in the dilution system is listed below:

12.1.2.1 Dilution Pump. Model A-150 Kohmyhr Teflon positive displacement type, nonadjustable 150 cc/min. ±2.0 percent, or equivalent, per dilution stage. A 9:1 dilution of sample is accomplished by combining 150 cc of sample with 1350 cc of clean dry air as shown in Figure 15-2.

12.1.2.2 Valves. Three-way Teflon solenoid or manual type.

12.1.2.3 Tubing. Teflon tubing and fittings are used throughout from the sample probe to the GC/FPD to present an inert surface for sample gas.

12.1.2.4 Box. Insulated box, heated and maintained at 120° C, of sufficient dimensions to house dilution apparatus.

12.1.2.5 Flowmeters. Rotameters or equivalent to measure flow from 0 to 1500 ml/min. ±1 percent per dilution stage.

12.1.3.0 Gas Chromatograph.

12.1.3.1 Column—1.83 m (6 ft.) length of Teflon tubing, 2.16 mm (0.085 in.) inside diameter, packed with deactivated silica gel, or equivalent.

12.1.3.2 Sample Valve. Teflon six port gas sampling valve, equipped with a 1 ml sample loop actuated by compressed air (Figure 15-1).

12.1.3.3 Oven. For containing sample valve, stripper column and separation column. The oven should be capable of maintaining an elevated temperature ranging from ambient to 100° C, constant within ±1° C.

12.1.3.4 Temperature Monitor. Thermocouple pyrometer to measure column oven, detector, and exhaust temperature ±1° C.

12.1.3.5 Flow System. Gas metering system to measure sample flow, hydrogen flow, oxygen flow and nitrogen carrier gas flow.

12.1.3.6 Detector. Flame photometric detector.

12.1.3.7 Electrometer. Capable of full scale amplification of linear ranges of $10^{-9}$ to $10^{-4}$ amperes full scale.

12.1.3.8 Power Supply. Capable of delivering up to 750 volts.

12.1.3.9 Recorder. Compatible with the output voltage range of the electrometer.

12.1.4 Calibration. Permeation tube system (Figure 15-3).

12.1.4.1 Tube Chamber. Glass chamber of sufficient dimensions to house permeation tubes.

12.1.4.2 Mass Flowmeters. Two mass flowmeters in the range 0-3 l/min. and 0-10 l/min. to measure air flow over permeation tubes at ±2 percent. These flowmeters shall be cross-calibrated at the beginning of each test. Using a convenient flow rate in the measuring range of both flowmeters, set and monitor the flow rate of gas over the permeation tubes. Injection of calibration gas generated at this flow rate as measured by one flowmeter followed by injection of calibration gas at the same flow rate as measured by the other flowmeter should agree within the specified precision limits. If they do not, then there is a problem with the mass flow measurement. Each mass flowmeter shall be calibrated prior to the first test with a wet test meter and thereafter at least once each year.

12.1.4.3 Constant Temperature Bath. Capable of maintaining permeation tubes at certification temperature of 30° C within ±0.1° C.

12.2 Reagents.

12.2.1 Fuel. Hydrogen ($H_2$) prepurified grade or better.

12.2.2 Combustion Gas. Oxygen ($O_2$) research purity or better.

12.2.3 Carrier Gas. Nitrogen ($N_2$) prepurified grade or better.

12.2.4 Diluent. Air containing less than 0.5 ppm total sulfur compounds and less than 10 ppm each of moisture and total hydrocarbons, and filtered using MSA filters 46727 and 79030, or equivalent. Removal of sulfur compounds can be verified by injecting dilution air only, described in section 8.3.

12.2.5 Compressed Air. 60 psig for GC valve actuation.

12.2.6 Calibration Gases. Permeation tubes gravimetrically calibrated and certified at 30.0° C.

12.3 Operating Parameters. The operating parameters for the GC/FPD system are as follows: nitrogen carrier gas flow rate of 100 cc/min, exhaust temperature of 110° C, detector temperature 105° C, oven temperature of 40° C, hydrogen flow rate of 80 cc/minute, oxygen flow rate of 20 cc minute, and sample flow rate of 80 cc/minute

12.4 Analysis. The sample valve is actuated for 1 minute in which time an aliquot of diluted sample is injected onto the separation column. The valve is then deactivated for the remainder of analysis cycle in which time the sample loop is refilled and the separation column continues to be foreflushed. The elution time for each compound will be determined during calibration.

### *13. Bibliography*

13.1 O'Keeffe, A. E. and G. C. Ortman. "Primary Standards for Trace Gas Analysis." Anal. Chem. 38,760 (1966).

13.2 Stevens, R. K., A. E. O'Keeffe, and G. C. Ortman. "Absolute Calibration of a Flame Photometric Detector to Volatile Sulfur Compounds at Sub-Part-Per-Million Levels." Environmental Science and Technology 3:7 (July, 1969).

13.3 Mulick, J. D., R. K. Stevens, and R. Baumgardner. "An Analytical System Designed to Measure Multiple Malodorous Compounds Related to Kraft Mill Activities." Presented at the 12th Conference on Methods in Air Pollution and Industrial Hygiene Studies, University of Southern California, Los Angeles, Calif April 5-8, 1971.

13.4 Devonald, R. H., R. S. Serenius, and A. D. McIntyre. "Evaluation of the Flame Photometric Detector for Analysis of Sulfur Compounds." Pulp and Paper Magazine of Canada, 73,3 (March, 1972).

13.5 Grimley, K. W., W. S. Smith, and R. M. Martin. "The Use of a Dynamic Dilution System in the Conditioning of Stack Gases for Automated Analysis by a Mobile Sampling Van." Presented at the 63rd Annual APCA Meeting in St. Louis, Mo. June 14-19, 1970.

13.6 General Reference. Standard Methods of Chemical Analysis Volume III A and B Instrumental Methods. Sixth Edition. Van Nostrand Reinhold Co.

# METHOD 16

## SEMICONTINUOUS DETERMINATION OF SULFUR EMISSIONS FROM STATIONARY SOURCES

### *Introduction*

The method described below uses the principle of gas chromatographic separation and flame photometric detection. Since there are many systems or sets of operating conditions that represent usable methods of determining sulfur emissions, all systems which employ this principle, but differ only in details of equipment and operation, may be used as alternative methods, provided that the criteria set below are met.

1. *Principle and Applicability.*

1.1 Principle. A gas sample is extracted from the emission source and diluted with clean dry air. An aliquot of the diluted sample is then analyzed for hydrogen sulfide ($H_2S$), methyl mercaptan (MeSH), dimethyl sulfide (DMS) and dimethyl disulfide (DMDS) by gas chromatographic (GC) separation and flame photometric detection (FPD). These four compounds are known collectively as total reduced sulfur (TRS).

1.2 Applicability. This method is applicable for determination of TRS compounds from recovery furnaces, lime kilns, and smelt dissolving tanks at kraft pulp mills.

2. *Range and Sensitivity.*

2.1 Range. Coupled with a gas chromatographic system utilizing a ten milliliter sample size, the maximum limit of the FPD for each sulfur compound is approximately 1 ppm. This limit is expanded by dilution of the sample gas before analysis. Kraft mill gas samples are normally diluted tenfold (9:1), resulting in an upper limit of about 10 ppm for each compound.

For sources with emission levels between 10 and 100 ppm, the measuring range can be best extended by reducing the sample size to 1 milliliter.

2.2 Using the sample size, the minimum detectable concentration is approximately 50 ppb.

3. *Interferences.*

3.1 Moisture Condensation. Moisture condensation in the sample delivery system, the analytical column, or the FPD burner block can cause losses or interferences. This potential is eliminated by heating the sample line, and by conditioning the sample with dry dilution air to lower its dew point below the operating temperature of the GC/FPD analytical system prior to analysis.

3.2 Carbon Monoxide and Carbon Dioxide. CO and $CO_2$ have substantial desensitizing effect on the flame photometric detector even after 9:1 dilution. Acceptable systems must demonstrate that they have eliminated this interference by some procedure such as eluting these compounds before any of the compounds to be measured. Compliance with this requirement can be demonstrated by submitting chromatograms of calibration gases with and without $CO_2$ in the diluent gas. The $CO_2$ level should be approximately 10 percent for the case with $CO_2$ present. The two chromatographs should show agreement within the precision limits of Section 4.1.

3.3 Particulate Matter. Particulate matter in gas samples can cause interference by eventual clogging of the analytical system. This interference must be eliminated by use of a probe filter.

3.4 Sulfur Dioxide. $SO_2$ is not a specific interferent but may be present in such large amounts that it cannot be effectively separated from other compounds of interest. The procedure must be designed to eliminate this problem either by the choice of separation columns or by removal of $SO_2$ from the sample.

Compliance with this section can be demonstrated by submitting chromatographs of calibration gases with $SO_2$ present in the same quantities expected from the emission source to be tested. Acceptable systems shall show baseline separation with the amplifier attenuation set so that the reduced sulfur compound of concern is at least 50 percent of full scale. Base line separation is defined as a return to zero ± percent in the interval between peaks.

4. *Precision and Accuracy.*

4.1 GC/FPD and Dilution System Calibration Precision. A series of three consecutive injections of the same calibration gas, at any dilution, shall produce results which do not vary by more than ±3 percent from the mean of the three injections.

4.2 GC/FPD and Dilution System Calibration Drift. The calibration drift determined from the mean of three injections made at the beginning and end of any 8-hour period shall not exceed ± percent.

4.3 System Calibration Accuracy. The complete system must quantitatively transport and analyze with an accuracy of 20 percent. A correction factor is developed to adjust calibration accuracy to 100 percent.

5. *Apparatus* (See Figure 16-1).

5.1.1 Probe. The probe must be made of inert material such as stainless steel or glass. It should be designed to incorporate a filter and to allow calibration gas to enter the probe at or near the sample entry point. Any portion of the probe not exposed to the stack gas must be heated to prevent moisture condensation.

5.1.2 Sample Line. The sample line must be made of Teflon,[1] no greater than 1.3 cm (½) inside diameter. All parts from the probe to the dilution system must be thermostatically heated to 120° C.

5.1.3 Sample Pump. The sample pump shall be a leakless Teflon-coated diaphragm type or equivalent. If the pump is upstream of the dilution system, the pump head must be heated to 120° C.

---

[1] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

5.2 Dilution System. The dilution system must be constructed such that all sample contacts are made of inert materials (e.g., stainless steel or Teflon). It must be heated to 120° C. and be capable of approximately a 9:1 dilution of the sample.

5.3 Gas Chromatograph. The gas chromatograph must have at least the following components:

5.3.1 Oven. Capable of maintaining the separation column at the proper operating temperature ±1° C.

5.3.2 Temperature Gauge. To monitor column oven, detector, and exhaust temperature ±1° C.

5.3.3 Flow System. Gas metering system to measure sample, fuel, combustion gas, and carrier gas flows.

5.3.4 Flame Photometric Detector.

5.3.4.1 Electrometer. Capable of full scale amplification of linear ranges of $10^{-9}$ to $10^{-4}$ amperes full scale.

5.3.4.2 Power Supply. Capable of delivering up to 750 volts.

5.3.4.3 Recorder. Compatible with the output voltage range of the electrometer.

5.4 Gas Chromatograph Columns. The column system must be demonstrated to be capble of resolving the four major reduced sulfur compounds: $H_2S$, MeSH, DMS, and DMDS. It must also demonstrate freedom from known interferences.

To demonstrate that adequate resolution has been achieved, the tester must submit a chromatograph of a calibration gas containing all four of the TRS compounds in the concentration range of the applicable standard. Adequate resolution will be defined as base line separation of adjacent peaks when the amplifier attenuation is set so that the smaller peak is at least 50 percent of full scale. Base line separation is defined in Section 3.4. Systems not meeting this criteria may be considered alternate methods subject to the approval of the Administrator.

5.5. Calibration System. The calibration system must contain the following components.

5.5.1 Tube Chamber. Chamber of glass or Teflon of sufficient dimensions to house permeation tubes.

5.5.2 Flow System. To measure air flow over permeation tubes at ±2 percent. Each flowmeter shall be calibrated after a complete test series with a wet test meter. If the flow measuring device differs from the wet test meter by 5 percent, the completed test shall be discarded. Alternatively, the tester may elect to use the flow data that would yield the lowest flow measurement. Calibration with a wet test meter before a test is optional.

5.5.3 Constant Temperature Bath. Device capable of maintaining the permeation tubes at the calibration temperature within ±0.1° C.

5.5.4 Temperature Gauge. Thermometer or equivalent to monitor bath temperature within ±1° C.

6. *Reagents.*

6.1 Fuel. Hydrogen ($H_2$) prepurified grade or better.

6.2 Combustion Gas. Oxygen ($O_2$) or air, research purity or better.

6.3 Carrier Gas. Prepurified grade or better.

6.4 Diluent. Air containing less than 50 ppb total sulfur compounds and less than 10 ppm each of moisture and total hydrocarbons. This gas must be heated prior to mixing with the sample to avoid water condensation at the point of contact.

6.5 Calibration Gases. Permeation tubes, one each of $H_2S$, MeSH, DMS, and DMDS, agravimetrically calibrated and certified at some convenient operating temperature. These tubes consist of hermetically sealed FEP Teflon tubing in which a liquified gaseous substance is enclosed. The enclosed gas permeates through the tubing wall at a constant rate. When the temperature is constant, calibration gases covering a wide range of known concentrations can be generated by varying and accurately measuring the flow rate of diluent gas passing over the tubes. These calibration gases are used to calibrate the GC/FPD system and the dilution system.

7. *Pretest Procedures.* The following procedures are optional but would be helpful in preventing any problem which might occur later and invalidate the entire test.

7.1 After the complete measurement system has been set up at the site and deemed to be operational, the following procedures should be completed before sampling is initiated.

7.1.1 Leak Test. Appropriate leak test procedures should be employed to verify the integrity of all components, sample lines, and connections. The following leak test procedure is suggested: For components upstream of the sample pump, attach the probe end of the sample line to a manometer or vacuum gauge, start the pump and pull greater than 50 mm (2 in.) Hg vacuum, close off the pump outlet, and then stop the pump and ascertain that there is no leak for 1 minute. For components after the pump, apply a slight positive pressure and check for leaks by applying a liquid (detergent in water, for example) at each joint. Bubbling indicates the presence of a leak.

7.1.2 System Performance. Since the complete system is calibrated following each test, the precise calibration of each component is not critical. However, these components should be verified to be operating properly. This verification can be performed by observing the response of flowmeters or of the GC output to changes in flow rates or calibration gas concentrations and ascertaining the response to be within predicted limits. In any component, or if the complete system fails to respond in a normal and predictable manner, the source of the discrepancy should be identified and corrected before proceeding.

8. *Calibration.* Prior to any sampling run, calibrate the system using the following procedures. (If more than one run is performed during any 24-hour period, a calibration need not be performed prior to the second and any subsequent runs. The calibration must, however, be verified as prescribed in Section 10, after the last run made within the 24-hour period.)

8.1 General Considerations. This section outlines steps to be followed for use of the GC/FPD and the dilution system. The procedure does not include detailed instructions because the operation of these systems is complex, and it requires a understanding of the individual system being used. Each system should include a written operating manual describing in detail the operating procedures associated with each component in the measurement system. In addition, the operator should be familiar with the operating principles of the components; particularly the GC/FPD. The citations in the Bibliography at the end of this method are recommended for review for this purpose.

8.2 Calibration Procedure. Insert the permeation tubes into the tube chamber.

Reproduced from
best available copy.

Check the bath temperature to assure agreement with the calibration temperature of the tubes within ±0.1° C. Allow 24 hours for the tubes to equilibrate. Alternatively equilibration may be verified by injecting samples of calibration gas at 1-hour intervals. The permeation tubes can be assumed to have reached equilibrium when consecutive hourly samples agree within the precision limits of Section 4.1.

Vary the amount of air flowing over the tubes to produce the desired concentrations for calibrating the analytical and dilution systems. The air flow across the tubes must at all times exceed the flow requirement of the analytical systems. The concentration in parts per million generated by a tube containing a specific permeant can be calculated as follows:

$$C = K \frac{P_r}{ML}$$

Equation 16-1

where:

C = Concentration of permeant produced in ppm.
$P_r$ = Permeation rate of the tube in μg/min.
M = Molecular weight of the permeant (g/g-mole).
L = Flow rate, 1/min, of air over permeant @ 20° C, 760 mm Hg.
K = Gas constant at 20° C and 760 mm Hg = 24.04 1/g mole.

8.3 Calibration of analysis system. Generate a series of three or more known concentrations spanning the linear range of the FPD (approximately 0.05 to 1.0 ppm) for each of the four major sulfur compounds. Bypassing the dilution system, inject these standards into the GC/FPD analyzers and monitor the responses. Three injects for each concentration must yield the precision described in Section 4.1. Failure to attain this precision is an indication of a problem in the calibration or analytical system. Any such problem must be identified and corrected before proceeding.

8.4 Calibration Curves. Plot the GC/FPD response in current (amperes) versus their causative concentrations in ppm on log-log coordinate graph paper for each sulfur compound. Alternatively, a least squares equation may be generated from the calibration data.

8.5 Calibration of Dilution System. Generate a known concentration of hydrogen sulfide using the permeation tube system. Adjust the flow rate of diluent air for the first dilution stage so that the desired level of dilution is approximated. Inject the diluted calibration gas into the GC/FPD system and monitor its response. Three injections for each dilution must yield the precision described in Section 4.1. Failure to attain this precision in this step is an indication of a problem in the dilution system. Any such problem must be identified and corrected before proceeding. Using the calibration data for $H_2S$ (developed under 8.3) determine the diluted calibration gas concentration in ppm. Then calculate the dilution factor as the ratio of the calibration gas concentration before dilution to the diluted calibration gas concentration determined under this paragraph. Repeat this procedure for each stage of dilution required. Alternatively, the GC/FPD system may be calibrated by generating a series of three or more concentrations of each sulfur compound and diluting these samples before injecting them into the GC/FPD system. This data will then serve as the calibration data for the unknown samples and a separate determination of the dilution factor will not be necessary. However, the precision requirements of Section 4.1 are still applicable.

9. *Sampling and Analysis Procedure.*

9.1 Sampling. Insert the sampling probe into the test port making certain that no dilution air enters the stack through the port. Begin sampling and dilute the sample approximtely 9:1 using the dilution system. Note that the precise dilution factor is that which is determined in paragraph 8.5. Condition the entire system with sample for a minimum of 15 minutes prior to commencing analysis.

9.2 Analysis. Aliquots of diluted sample are injected into the GC/FPD analyzer for analysis.

9.2.1 Sample Run. A sample run is composed of 16 individual analyses (injects) performed over a period of not less than 3 hours or more than 6 hours.

9.2.2 Observation for Clogging of Probe. If reductions in sample concentrations are observed during a sample run that cannot be explained by process conditions, the sampling must be interrupted to determine if the sample probe is clogged with particulate matter. If the probe is found to be clogged, the test must be stopped and the results up to that point discarded. Testing may resume after cleaning the probe or replacing it with a clean one. After each run, the sample probe must be inspected and, if necessary, dismantled and cleaned.

10. *Post-Test Procedures.*

10.1 Sample Line Loss. A known concentration of hydrogen sulfide at the level of the applicable standard, ±20 percent, must be introduced into the sampling system at the opening of the probe in sufficient quantities to insure that there is an excess of sample which must be vented to the atmosphere. The sample must be transported through the entire sampling system to the measurement system in the normal manner. The resulting measured concentration should be compared to the known value to determine the sampling system loss. A sampling system loss of more than 20 percent is unacceptable. Sampling losses of 0-20 percent must be corrected for by dividing the resulting sample concentration by the fraction of recovery. The known gas sample may be generated using permeation tubes. Alternatively, cylinders of hydrogen sulfide mixed in air may be used provided they are traceable to permeation tubes. The optional pretest procedures provide a good guideline for determining if there are leaks in the sampling system.

10.2 Recalibration. After each run, or after a series of runs made within a 24-hour period, perform a partial recalibration using the procedures in Section 8. Only $H_2S$ (or other permeant) need be used to recalibrate the GC/FPD analysis system (8.3) and the dilution system (8.5).

10.3 Determination of Calibration Drift. Compare the calibration curves obtained prior to the runs, to the calibration curves obtained under paragraph 10.1. The calibration drift should not exceed the limits set forth in paragraph 4.2. If the drift exceeds this limit, the intervening run or runs should be considered not valid. The tester, however, may instead have the option of choosing the calibration data set which would give the highest sample values.

11. *Calculations.*

11.1 Determine the concentrations of each reduced sulfur compound detected directly from the calibration curves. Alternatively, the concentrations may be calculated using the equation for the least square line.

11.2 Calculation of TRS. Total reduced sulfur will be determined for each anaylsis made by summing the concentrations of each reduced sulfur compound resolved during a given analysis.

$$TRS = \Sigma\,(H_2S,\ MeSH,\ DMS,\ 2DMDS)d$$

Equation 16-2

where:

TRS = Total reduced sulfur in ppm, wet basis.
$H_2S$ = Hydrogen sulfide, ppm.
MeSH = Methyl mercaptan, ppm.
DMS = Dimethyl sulfide, ppm.
DMDS = Dimethyl disulfide, ppm.
d = Dilution factor, dimensionless.

11.3 Average TRS. The average TRS will be determined as follows:

$$\text{Average TRS} = \frac{\sum_{i=1}^{N} TRS_i}{N(1-B_{wo})}$$

Average TRS = Average total reduced suflur in ppm, dry basis
$TRS_i$ = Total reduced sulfur in ppm as determined by Equation 16-2.
N = Number of samples.
$B_{wo}$ = Fraction of volume of water vapor in the gas stream as determined by method 4—Determination of Moisture in Stack Gases (36 FR 24887).

11.4 Average concentration of individual reduced sulfur compounds.

$$C = \frac{\sum_{i=1}^{N} S_i}{N}$$

Equation 16-3

where:

$S_i$ = Concentration of any reduced sulfur compound from the ith sample injection, ppm.
C = Average concentration of any one of the reduced sulfur compounds for the entire run, ppm.
N = Number of injections in any run period.

12. *Example System.* Described below is a system utilized by EPA in gathering NSPS data. This system does not now reflect all the latest developments in equipment and column technology, but it does represent one system that has been demonstrated to work.

12.1 Apparatus.

12.1.1 Sampling System.

12.1.1.1 Probe. Figure 16-1 illustrates the probe used in lime kilns and other sources where significant amounts of particulate matter are present. the probe is designed with the deflector shield placed between the sample and the gas inlet holes and the glass wool plugs to reduce clogging of the filter and possible adsorption of sample gas. The exposed portion of the probe between the sampling port and the sample line is heated with heating tape.

12.1.1.2 Sample Line $\frac{3}{16}$ inch inside diameter Teflon tubing, heated to 120° C. This temperature is controlled by a thermostatic heater.

12.1.1.3 Sample Pump. Leakless Teflon coated diaphragm type or equivalent. The pump head is heated to 120° C by enclosing it in the sample dilution box (12.2.4 below).

12.1.2 Dilution System. A schematic diagram of the dynamic dilution system is given in Figure 16-2. The dilution system is constructed such that all sample contacts are made of inert materials. The dilution system which is heated to 120° C must be capable of a minimum of 9:1 dilution of sample. Equipment used in the dilution system is listed below:

12.1.2.1 Dilution Pump. Model A-150 Kohmyhr Teflon positive displacement type, nonadjustable 150 cc/min. ±2.0 percent, or equivalent, per dilution stage. A 9:1 dilution of sample is accomplished by combining 150 cc of sample with 1,350 cc of clean dry air as shown in Figure 16-2.

12.1.2.2 Valves. Three-way Teflon solenoid or manual type.

12.1.2.3 Tubing. Teflon tubing and fittings are used throughout from the sample probe to the GC/FPD to present an inert surface for sample gas.

12.1.2.4 Box. Insulated box, heated and maintained at 120° C, of sufficient dimensions to house dilution apparatus.

12.1.2.5 Flowmeters. Rotameters or equivalent to measure flow from 0 to 1500 ml/min ±1 percent per dilution stage.

12.1.3 Gas Chromatograph Columns. Two types of columns are used for separation of low and high molecular weight sulfur compounds:

12.1.3.1 Low Molecular Weight Sulfur Compounds Column (GC/FPD-1).

12.1.3.1 Separation Column. 11 m by 2.16 mm (36 ft by 0.085 in) inside diameter Teflon tubing packed with 30/60 mesh Teflon coated with 5 percent polyphenyl ether and 0.05 percent orthophosphoric acid, or equivalent (see Figure 16-3).

12.1.3.1.2 Stripper or Precolumn. 0.6 m by 2.16 mm (2 ft by 0.085 in) inside diameter Teflon tubing packed as in 5.3.1.

12.1.3.1.3 Sample Valve. Teflon 10-port gas sampling valve, equipped with a 10 ml sample loop, actuated by compressed air (Figure 16-3).

12.1.3.1.4 Oven. For containing sample valve, stripper column and separation column. The oven should be capable of maintaining an elevated temperature ranging from ambient to 100° C, constant within ±1° C.

12.1.3.1.5 Temperature Monitor. Thermocouple pyrometer to measure column oven, detector, and exhaust temperature ±1° C.

12.1.3.1.6 Flow System. Gas metering system to measure sample flow, hydrogen flow, and oxygen flow (and nitrogen carrier gas flow).

12.1.3.1.7 Detector. Flame photometric detector.

12.1.3.1.8 Electrometer. Capable of full scale amplification of linear ranges of $10^{-9}$ to $10^{-4}$ amperes full scale.

12.1.3.1.9 Power Supply. Capable of delivering up to 750 volts.

12.1.3.1.10 Recorder. Compatible with

the output voltage range of the electrometer.

12.1.3.2 High Molecular Weight Compounds Column (GC/FPD-11).

12.1.3.2.1. Separation Column. 3.05 m by 2.16 mm (10 ft by 0.0885 in) inside diameter Teflon tubing packed with 30/60 mesh Teflon coated with 10 percent Triton X-305, or equivalent.

12.1.3.2.2 Sample Valve. Teflon 6-port gas sampling valve equipped with a 10 ml sample loop, actuated by compressed air (Figure 16-3).

12.1.3.2.3 Other Components. All components same as in 12.1.3.1.4 to 12.1.3.1.10.

12.1.4 Calibration. Permeation tube system (figure 16-4).

12.1.4.1 Tube Chamber. Glass chamber of sufficient dimensions to house permeation tubes.

12.1.4.2 Mass Flowmeters. Two mass flowmeters in the range 0-3 1/min. and 0-10 1/min. to measure air flow over permeation tubes at ± 2 percent. These flowmeters shall be cross-calibrated at the beginning of each test. Using a convenient flow rate in the measuring range of both flowmeters, set and monitor the flow rate of gas over the permeation tubes. Injection of calibration gas generated at this flow rate as measured by one flowmeter followed by injection of calibration gas at the same flow rate as measured by the other flowmeter should agree within the specified precision limits. If they do not, then there is a problem with the mass flow measurement. Each mass flowmeter shall be calibrated prior to the first test with a wet test meter and thereafter, at least once each year.

12.1.4.3 Constant Temperature Bath. Capable of maintaining permeation tubes at certification temperature of 30° C. within ±0.1° C.

12.2 Reagents

12.2.1 Fuel. Hydrogen ($H_2$) prepurified grade or better.

12.2.2. Combustion Gas. Oxygen ($O_2$) research purity or better.

12.2.3 Carrier Gas. Nitrogen ($N_2$) prepurified grade or better.

12.2.4 Diluent. Air containing less than 50 ppb total sulfur compounds and less than 10 ppm each of moisture and total hydrocarbons, and filtered using MSA filters 46727 and 79030, or equivalent. Removal of sulfur compounds can be verified by injecting dilution air only, described in Section 8.3.

12.2.5 Compressed Air. 60 psig for GC valve actuation.

12.2.6 Calibrated Gases. Permeation tubes gravimetrically calibrated and certified at 30.0° C.

12.3 Operating Parameters.

12.3.1 Low-Molecular Weight Sulfur Compounds. The operating parameters for the GC/FPD system used for low molecular weight compounds are as follows: nitrogen carrier gas flow rate of 50 cc/min, exhaust temperature of 110° C, detector temperature of 105° C, oven temperature of 40° C, hydrogen flow rate of 80 cc/min, oxygen flow rate of 20 cc/min, and sample flow rate between 20 and 80 cc/min.

12.3.2 High-Molecular Weight Sulfur Compounds. The operating parameters for the GC/FPD system for high molecular weight compounds are the same as in 12.3.1 except: oven temperature of 70° C, and nitrogen carrier gas flow of 100 cc/min.

12.4 Analysis Procedure.

12.4.1 Analysis. Aliquots of diluted sample are injected simultaneously into both GC/FPD analyzers for analysis. GC/FPD-I is used to measure the low-molecular weight reduced sulfur compounds. The low molecular weight compounds include hydrogen sulfide, methyl mercaptan, and dimethyl sulfide. GC/FPD-II is used to resolve the high-molecular weight compound. The high-molecular weight compound is dimethyl disulfide.

12.4.1.1 Analysis of Low-Molecular Weight Sulfur Compounds. The sample valve is actuated for 3 minutes in which time an aliquot of diluted sample is injected into the stripper column and analytical column. The valve is then deactivated for approximately 12 minutes in which time, the analytical column continues to be foreflushed, the stripper column is backflushed, and the sample loop is refilled. Monitor the responses. The elution time for each compound will be determined during calibration.

12.4.1.2 *Analysis of High-Molecular Weight Sulfur Compounds. The procedure is essentially the same as above except that no stripper column is needed.

13. *Bibliography.*

13.1 O'Keeffe, A. E. and G. C. Ortman. "Primary Standards for Trace Gas Analysis." Analytical Chemical Journal, 38,760 (1966).

13.2 Stevens, R. K., A. E. O'Keeffe, and G. C. Ortman. "Absolute Calibration of a Flame Photometric Detector to Volatile Sulfur Compounds at Sub-Part Per-Million Levels." Environmental Science and Technology, 3:7 (July, 1969).

13.3 Mulick, J. D., R. K. Stevens, and R. Baumgardner. "An Analytical System Designed to Measure Multiple Malodorous Compounds Related to Kraft Mill Activities." Presented at the 12th Conference on Methods in Air Pollution and Industrial Hygiene Studies, University of Southern California, Los Angeles, CA. April 6-8, 1971.

13.4 Devonald, R. H., R. S. Serenius, and A. D. McIntyre. "Evaluation of the Flame Photometric Detector for Analysis of Sulfur Compounds." Pulp and Paper Magazine of Canada, 73,3 (March, 1972).

13.5 Grimley, K. W., W. S. Smith, and R. M. Martin. "The Use of a Dynamic Dilution System in the Conditioning of Stack Gases for Automated Analysis by a Mobile Sampling Van." Presented at the 63rd Annual APCA Meeting in St. Louis, Mo. June 14-19, 1970.

13.6 General Reference. Standard Methods of Chemical Analysis Volume III A and B Instrumental Methods. Sixth Edition. Van Nostrand Reinhold Co.

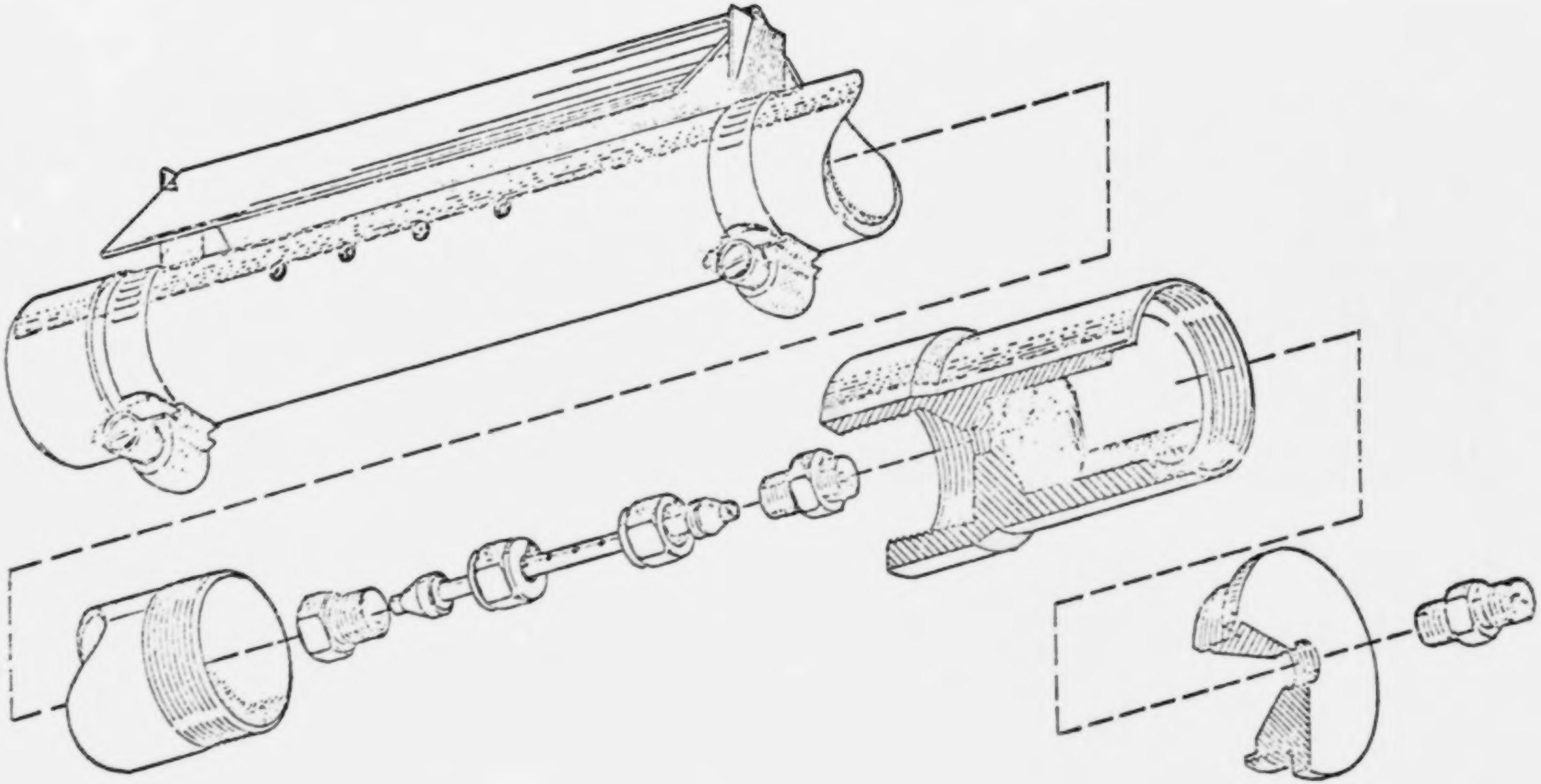


Figure 16-1. Probe used for sample gas containing high particulate loadings.

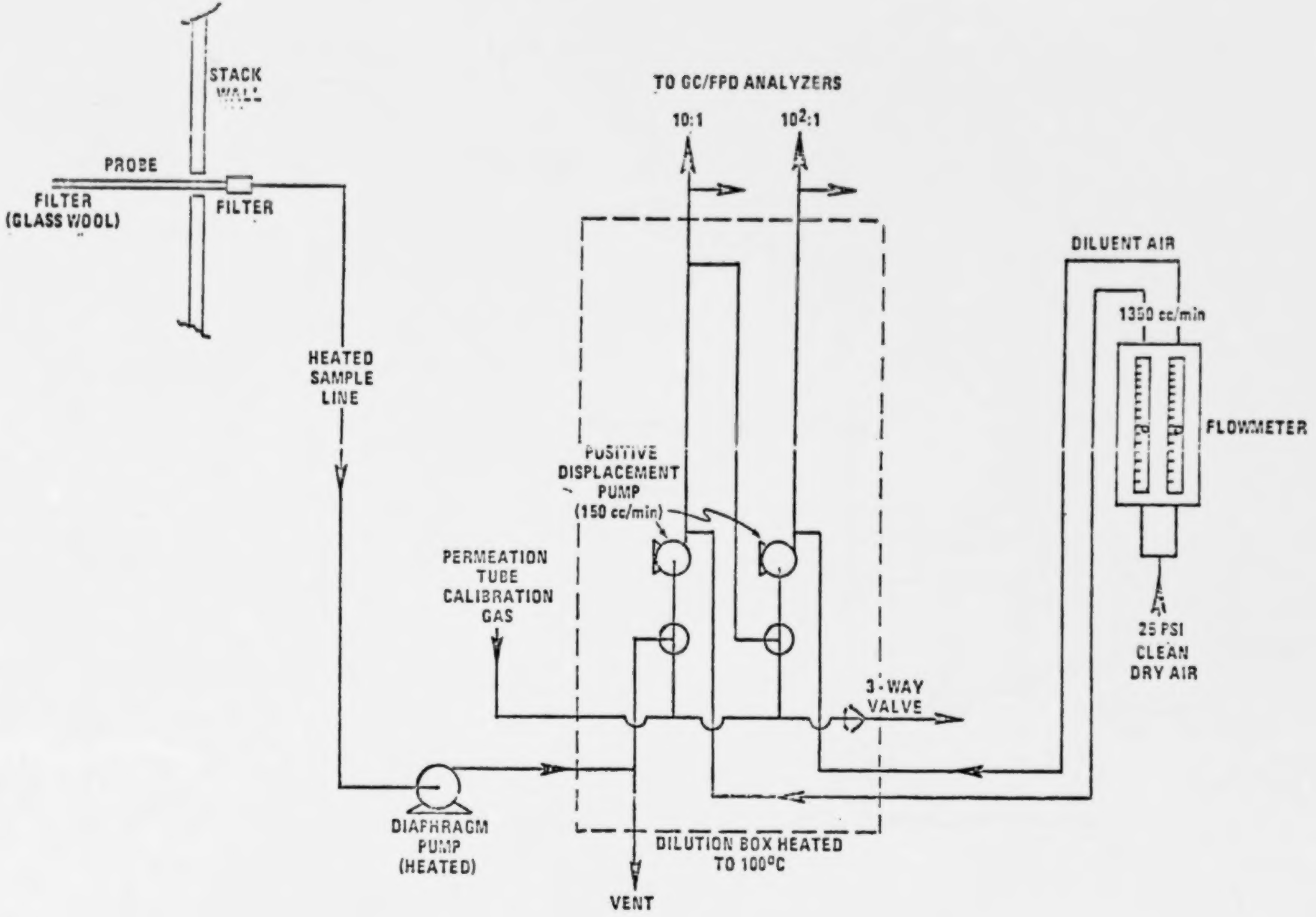

STACK WALL
PROBE
FILTER (GLASS WOOL)
FILTER
HEATED SAMPLE LINE
TO GC/FPD ANALYZERS
10:1
10²:1
DILUENT AIR
1350 cc/min
FLOWMETER
POSITIVE DISPLACEMENT PUMP (150 cc/min)
PERMEATION TUBE CALIBRATION GAS
25 PSI CLEAN DRY AIR
3-WAY VALVE
DIAPHRAGM PUMP (HEATED)
DILUTION BOX HEATED TO 100°C
VENT

Figure 16-2. Sampling and dilution apparatus.

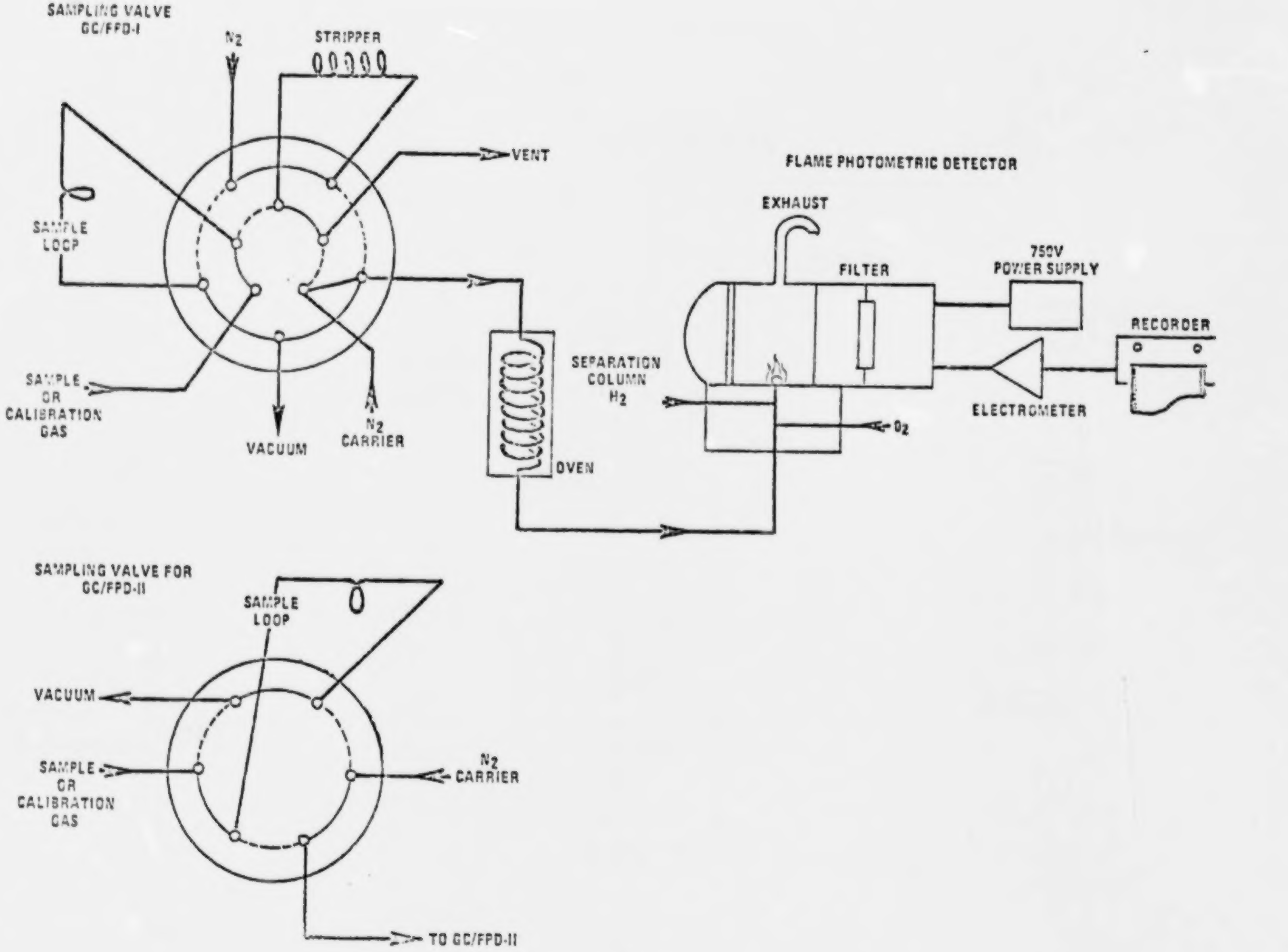

SAMPLING VALVE
GC/FPD-I
$N_2$
STRIPPER
VENT
SAMPLE
LOOP
SAMPLE
OR
CALIBRATION
GAS
VACUUM
$N_2$
CARRIER
SEPARATION
COLUMN
$H_2$
OVEN
FLAME PHOTOMETRIC DETECTOR
EXHAUST
FILTER
750V
POWER SUPPLY
RECORDER
ELECTROMETER
$O_2$
SAMPLING VALVE FOR
GC/FPD-II
SAMPLE
LOOP
VACUUM
SAMPLE
OR
CALIBRATION
GAS
$N_2$
CARRIER
TO GC/FPD-II

Figure 16-3. Gas chromatographic-flame photometric analyzers.

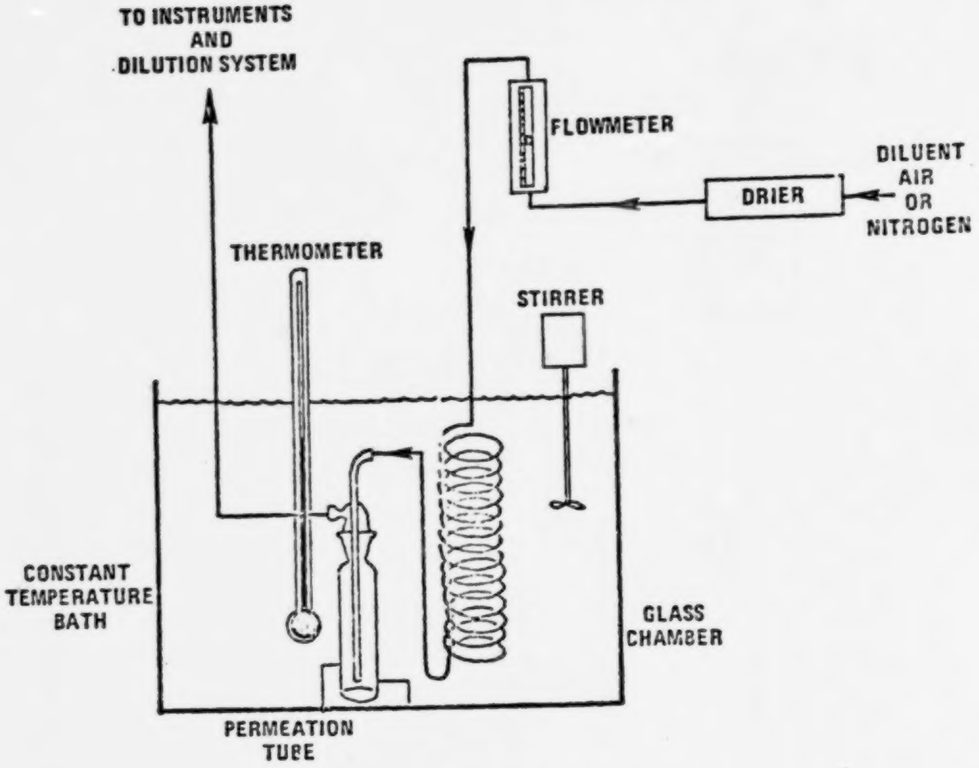

TO INSTRUMENTS AND DILUTION SYSTEM
FLOWMETER
DILUENT AIR OR NITROGEN
DRIER
THERMOMETER
STIRRER
CONSTANT TEMPERATURE BATH
GLASS CHAMBER
PERMEATION TUBE

Figure 16-4. Apparatus for field calibration.

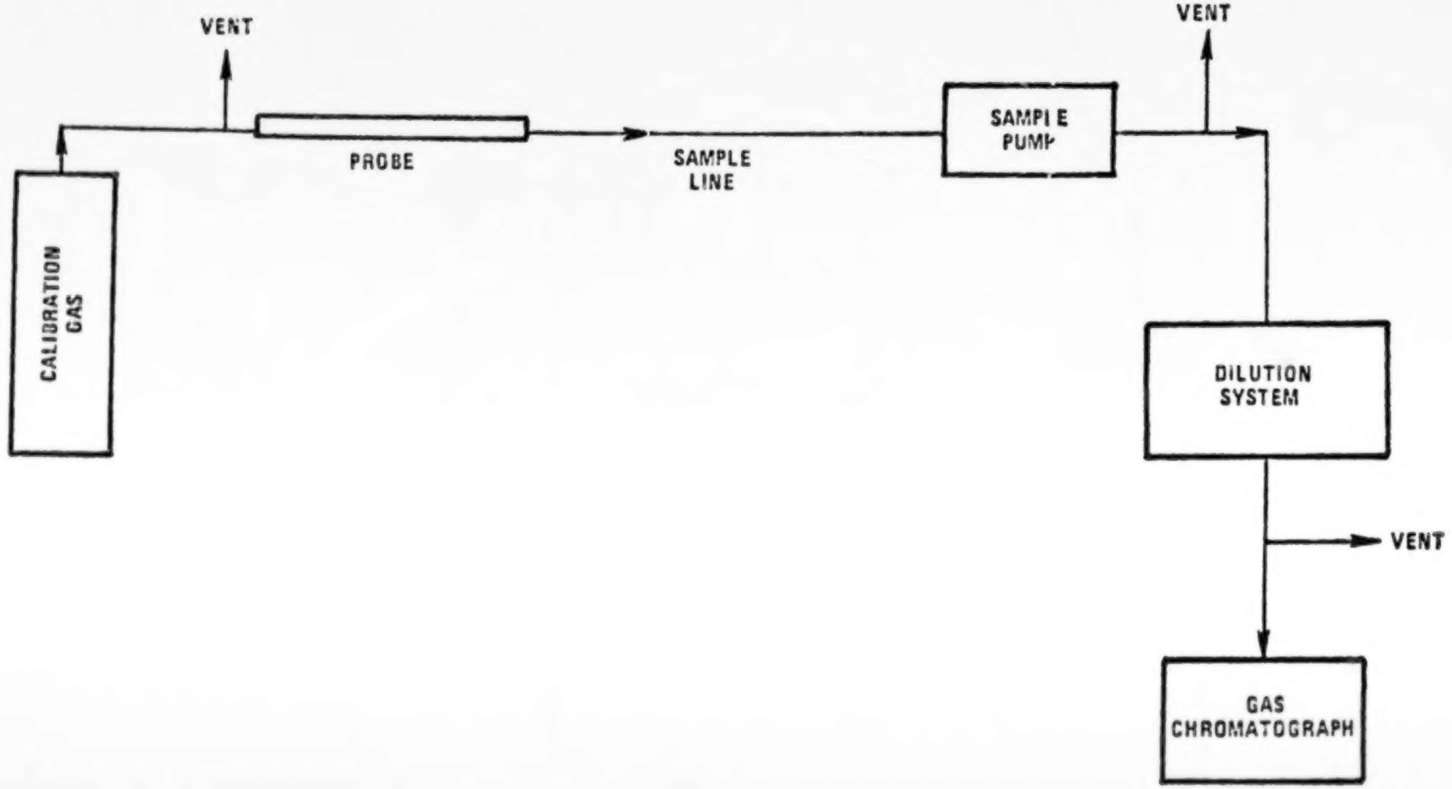

VENT
CALIBRATION GAS
PROBE
SAMPLE LINE
SAMPLE PUMP
VENT
DILUTION SYSTEM
VENT
GAS CHROMATOGRAPH

Figure 16- 5. Determination of sample line loss.

# METHOD 17

# DETERMINATION OF PARTICULATE EMISSIONS FROM STATIONARY SOURCES (IN-STACK FILTRATION METHOD)

### *Introduction*

Particulate matter is not an absolute quantity; rather, it is a function of temperature and pressure. Therefore, to prevent variability in particulate matter emission regulations and/or associated test methods, the temperature and pressure at which particulate matter is to be measured must be carefully defined. Of the two variables (i.e., temperature and pressure), temperature has the greater effect upon the amount of particulate matter in an effluent gas stream; in most stationary source categories, the effect of pressure appears to be negligible.

In method 5, 250° F is established as a nominal reference temperature. Thus, where Method 5 is specified in an applicable subpart of the standards, particulate matter is defined with respect to temperature. In order to maintain a collection temperature of 250° F, Method 5 employs a heated glass sample probe and a heated filter holder. This equipment is somewhat cumbersome and requires care in its operation. Therefore, where particulate matter concentrations (over the normal range of temperature associated with a specified source category) are known to be independent of temperature, it is desirable to eliminate the glass probe and heating systems, and sample at stack temperature.

This method describes an in-stack sampling system and sampling procedures for use in such cases. It is intended to be used only when specified by an applicable subpart of the standards, and only within the applicable temperature limits (if specified), or when otherwise approved by the Administrator.

1. *Principle and Applicability.*

1.1 Principle. Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at stack temperature. The particulate mass is determined gravimetrically after removal of uncombined water.

1.2 Applicability. This method applies to the determination of particulate emissions from stationary sources for determining compliance with new source performance standards, only when specifically provided for in an applicable subpart of the standards. This method is not applicable to stacks that contain liquid droplets or are saturated with water vapor. In addition, this method shall not be used as written if the projected cross-sectional area of the probe extension-filter holder assembly covers more than 5 percent of the stack cross-sectional area (see Section 4.1.2).

2. *Apparatus.*

2.1 Sampling Train. A schematic of the sampling train used in this method is shown in Figure 17-1. Construction details for many, but not all, of the train components are given in APTD-0581 (Citation 2 in Section 7); for changes from the APTD-0581 document and for allowable modifications to Figure 17-1, consult with the Administrator.

The operating and maintenance procedures for many of the sampling train components are described in APTD-0576 (Citation 3 in Section 7). Since correct usage is important in obtaining valid results, all users should read the APTD-0576 document and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein. The sampling train consists of the following components:

2.1.1 Probe Nozzle. Stainless steel (316) or glass, with sharp, tapered leading edge. The angle of taper shall be 030° and the taper shall be on the outside to preserve a constant internal diameter. The probe nozzle shall be of the button-hook or elbow design, unless otherwise specified by the Administrator. If made of stainless steel, the nozzle shall be constructed from seamless tubing. Other materials of construction may be used subject to the approval of the Administrator.

A range of sizes suitable for isokinetic sampling should be available, e.g., 0.32 to 1.27 cm (⅛ to ½ in)—or larger if higher volume sampling trains are used—inside diameter (ID) nozzles in increments of 0.16 cm (1/16 in). Each nozzle shall be calibrated according to the procedures outlined in Section 5.1.

2.1.2 Filter Holder. The in-stack filter holder shall be constructed of borosilicate or quartz glass, or stainless steel; if a gasket is used, it shall be made of silicone rubber, Teflon, or stainless steel. Other holder and gasket materials may be used subject to the approval of the Administrator. The filter holder shall be designed to provide a positive seal against leakage from the outside or around the filter.

2.1.3 Probe Extension. Any suitable rigid probe extension may be used after the filter holder.

2.1.4 Pitot Tube. Type S, as described in Section 2.1 of Method 2, or other device approved by the Administrator; the pitot tube shall be attached to the probe extension to allow constant monitoring of the stack gas velocity (see Figure 17-1). The impact (high pressure) opening plane of the pitot tube shall be even with or above the nozzle entry plane during sampling (see Method 2, Figure 2-6b). It is recommended: (1) that the pitot tube have a known baseline coefficient, determined as outlined in Section 4 of Method 2; and (2) that this known coefficient be preserved by placing the pitot tube in an interference-free arrangement with respect to the sampling nozzle, filter holder, and temperature sensor (see Figure 17-1). Note that the 1.9 cm (0.75 in) free-space between the nozzle and pitot tube shown in Figure 17-1, is based on a 1.3 cm (0.5 in) ID nozzle. If the sampling train is designed for sampling at higher flow rates than that described in APTD-0581, thus necessitating the use of larger sized nozzles, the free-space shall be 1.9 cm (0.75 in) with the largest sized nozzle in place.

Source sampling assemblies that do not meet the minimum spacing requirements of Figure 17-1 (or the equivalent of these requirements, e.g., Figure 2-7 of Method 2) may be used; however, the pitot tube coefficients of such assemblies shall be determined by calibration, using methods subject to the approval of the Administrator.

2.1.5 Differential Pressure Gauge. Inclined manometer or equivalent device (two), as described in Section 2.2 of Method 2. One manometer shall be used for velocity head (Δp) readings, and the other, for orifice differential pressure readings.

2.1.6 Condenser. It is recommended that the impinger system described in Method 5 be used to determine the moisture content of the stack gas. Alternatively, any system that allows measurement of both the water condensed and the moisture leaving the condenser, each to within 1 ml or 1 g, may be used. The moisture leaving the condenser can be measured either by: (1) monitoring the temperature and pressure at the exit of the condenser and using Dalton's law of partial pressures, or (2) passing the sample gas stream through a silica gel trap with exit gases kept below 20° C (68° F) and determining the weight gain.

Flexible tubing may be used between the probe extension and condenser. If means other than silica gel are used to determine the amount of moisture leaving the condenser, it is recommended that silica gel still be used between the condenser system and pump to prevent moisture condensation in the pump and metering devices and to avoid the need to make corrections for moisture in the metered volume.

2.1.7 Metering System. Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3° C (5.4° F), dry gas meter capable of measuring volume to within 2 percent, and related equipment, as shown in Figure 17-1. Other metering systems capable of maintaining sampling rates within 10 percent of isokinetic and of determining sample volumes to within 2 percent may be used, subject to the approval of the Administrator. When the metering system is used in conjunction with a pitot tube, the system shall enable checks of isokinetic rates.

Sampling trains utilizing metering systems designed for higher flow rates than that described in APTD-0581 or APTD-0576 may be used provided that the specifications of this method are met.

2.1.8 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in Hg). In many cases, the barometric reading may be obtained from a nearby national weather service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

2.1.9 Gas Density Determination Equipment. Temperature sensor and pressure gauge, as described in Sections 2.3 and 2.4 of Method 2, and gas analyzer, if necessary, as described in Method 3.

The temperature sensor shall be attached to either the pitot tube or to the probe extension, in a fixed configuration. If the temperature sensor is attached in the field, the sensor shall be placed in an interference-free arrangement with respect to the Type S pitot tube openings (as shown in Figure 17-1 or in Figure 2-7 of Method 2). Alternatively, the temperature sensor need not be attached to either the probe extension or pitot tube during sampling, provided that a difference of not more than 1 percent in the average velocity measurement is introduced. This alternative is subject to the approval of the Administrator.

2.2 Sample Recovery.

2.2.1 Probe Nozzle Brush. Nylon bristle brush with stainless steel wire handle. The brush shall be properly sized and shaped to brush out the probe nozzle.

2.2.2 Wash Bottles—Two. Glass wash bottles are recommended; polyethylene wash bottles may be used at the option of the tester. It is recommended that acetone not be stored in polyethylene bottles for longer than a month.

2.2.3 Glass Sample Storage Containers. Chemically resistant, borosilicate glass bottles, for acetone washes, 500 ml or 1000 ml. Screw cap liners shall either be rubber-backed Teflon or shall be constructed so as to be leak-free and resistant to chemical attack by acetone. (Narrow mouth glass bottles have been found to be less prone to leakage.) Alternatively, polyethylene bottles may be used.

2.2.4 Petri Dishes. For filter samples; glass or polyethylene, unless otherwise specified by the Administrator.

2.2.5 Graduated Cylinder and/or Balance. To measure condensed water to within 1 ml or 1 g. Graduated cylinders shall have subdivisions no greater than 2 ml. Most laboratory balances are capable of weighing to the nearest 0.5 g or less. Any of these balances is suitable for use here and in Section 2.3.4.

2.2.6 Plastic Storage Containers. Air tight containers to store silica gel.

2.2.7 Funnel and Rubber Policeman. To aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

2.2.8 Funnel. Glass or polyethylene, to aid in sample recovery.

2.3 Analysis.

2.3.1 Glass Weighing Dishes.

2.3.2 Desiccator.

2.3.3 Analytical Balance. To measure to within 0.1 mg.

2.3.4 Balance. To measure to within 0.5 mg.

2.3.5 Beakers. 250 ml.

2.3.6 Hygrometer. To measure the relative humidity of the laboratory environment.

2.3.7 Temperature Gauge. To measure the temperature of the laboratory environment.

3. *Reagents.*

3.1 Sampling.

3.1.1 Filters. The in-stack filters shall be glass mats or thimble fiber filters, without organic binders, and shall exhibit at least 99.95 percent efficiency (00.05 percent penetration) on 0.3 micron dioctyl phthalate

Reproduced from
best available copy.

smoke particles. The filter efficiency tests shall be conducted in accordance with ASTM standard method D 2986-71. Test data from the supplier's quality control program are sufficient for this purpose.

3.1.2 Silica Gel. Indicating type, 6- to 16-mesh. If previously used, dry at 175° C (350° F) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to the approval of the Administrator.

3.1.3 Crushed Ice.

3.1.4 Stopcock Grease. Acetone-insoluble, heat-stable silicone grease. This is not necessary if screw-on connectors with Teflon sleeves, or similar, are used. Alternatively, other types of stopcock grease may be used, subject to the approval of the Administrator.

3.2 Sample Recovery. Acetone, reagent grade, 00.001 percent residue, in glass bottles. Acetone from metal containers generally has a high residue blank and should not be used. Sometimes, suppliers transfer acetone to glass bottles from metal containers. Thus, acetone blanks shall be run prior to field use and only acetone with low blank values (00.001 percent) shall be used. In no case shall a blank value of greater than 0.001 percent of the weight of acetone used be subtracted from the sample weight.

3.3 Analysis.

3.3.1 Acetone. Same as 3.2.

3.3.2 Desiccant. Anhydrous calcium sulfate, indicating type. Alternatively, other types of desiccants may be used, subject to the approval of the Administrator.

4. *Procedure.*

4.1 Sampling. The complexity of this method is such that, in order to obtain reliable results, testers should be trained and experienced with the test procedures.

4.1.1 Pretest Preparation. All components shall be maintained and calibrated according to the procedure described in APTD-0576, unless otherwise specified herein.

Weigh several 200 to 300 g portions of silica gel in air-tight containers to the nearest 0.5 g. Record the total weight of the silica gel plus container, on each container. As an alternative, the silica gel need not be preweighed, but may be weighed directly in its impinger or sampling holder just prior to train assembly.

Check filters visually against light for irregularities and flaws or pinhole leaks. Label filters of the proper size on the back side near the edge using numbering machine ink. As an alternative, label the shipping containers (glass or plastic petri dishes) and keep the filters in these containers at all times except during sampling and weighing.

Desiccate the filters at 20±5.6° C (68±10° F) and ambient pressure for at least 24 hours and weigh at intervals of at least 6 hours to a constant weight, i.e., 00.5 mg change from previous weighing; record results to the nearest 0.1 mg. During each weighing the filter must not be exposed to the laboratory atmosphere for a period greater than 2 minutes and a relative humidity above 50 percent. Alternatively (unless otherwise specified by the Administrator), the filters may be oven dried at 105° C (220° F) for 2 to 3 hours, desiccated for 2 hours, and weighed. Procedures other than those described, which account for relative humidity effects, may be used, subject to the approval of the Administrator.

4.1.2 Preliminary Determinations. Select the sampling site and the minimum number of sampling points according to Method 1 or as specified by the Administrator. Make a projected-area model of the probe extension-filter holder assembly, with the pitot tube face openings positioned along the centerline of the stack, as shown in Figure 17-2. Calculate the estimated cross-section blockage, as shown in Figure 17-2. If the blockage exceeds 5 percent of the duct cross sectional area, the tester has the following options: (1) a suitable out-of-stack filtration method may be used instead of in-stack filtration; or (2) a special in-stack arrangement, in which the sampling and velocity measurement sites are separate, may be used; for details concerning this approach, consult with the Administrator (see also Citation 10 in Section 7). Determine the stack pressure, temperature, and the range of velocity heads using Method 2; it is recommended that a leak-check of the pitot lines (see Method 2, Section 3.1) be performed. Determine the moisture content using Approximation Method 4 or its alternatives for the purpose of making isokinetic sampling rate settings. Determine the stack gas dry molecular weight, as described in Method 2, Section 3.6; if integrated Method 3 sampling is used for molecular weight determination, the integrated bag sample shall be taken simultaneously with, and for the same total length of time as the particular sample run.

Select a nozzle size based on the range of velocity heads, such that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle size. Ensure that the proper differential pressure gauge is chosen for the range of velocity heads encountered (see Section 2.2 of Method 2).

Select a probe extension length such that all traverse points can be sampled. For large stacks, consider sampling from opposite sides of the stack to reduce the length of probes.

Select a total sampling time greater than or equal to the minimum total sampling time specified in the test procedures for the specific industry such that (1) the sampling time per point is not less than 2 minutes (or some greater time interval if specified by the Administrator), and (2) the sample volume taken (corrected to standard conditions) will exceed the required minimum total gas sample volume. The latter is based on an approximate average sampling rate.

It is recommended that the number of minutes sampled at each point be an integer or an integer plus one-half minute, in order to avoid timekeeping errors.

In some circumstances, e.g., batch cycles, it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes. In these cases, the Administrator's approval must first be obtained.

4.1.3 Preparation of Collection Train. During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to begin.

If impingers are used to condense stack gas moisture, prepare them as follows: place 100 ml of water in each of the first two impingers, leave the third impinger empty, and transfer approximately 200 to 300 g of preweighed silica gel from its container to the fourth impinger. More silica gel may be used, but care should be taken to ensure that it is not entrained and carried out from the impinger during sampling. Place the container in a clean place for later use in the sample recovery. Alternatively, the weight of the silica gel plus impinger may be determined to the nearest 0.5 g and recorded.

If some means other than impingers is used to condense moisture, prepare the condenser (and, if appropriate, silica gel for condenser outlet) for use.

Using a tweezer or clean disposable surgical gloves, place a labeled (identified) and weighed filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as not to allow the sample gas stream to circumvent the filter. Check filter for tears after assembly is completed. Mark the probe extension with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

Assemble the train as in Figure 17-1, using a very light coat of silicone grease on all ground glass joints and greasing only the outer portion (see APTD-0576) to avoid possibility of contamination by the silicone grease. Place crushed ice around the impingers.

4.1.4 Leak Check Procedures.

4.1.4.1 Pretest Leak-Check. A pretest leak-check is recommended, but not required. If the tester opts to conduct the pretest leak-check, the following procedure shall be used.

After the sampling train has been assembled, plug the inlet to the probe nozzle with a material that will be able to withstand the stack temperature. Insert the filter holder into the stack and wait approximately 5 minutes (or longer, if necessary) to allow the system to come to equilibrium with the temperature of the stack gas stream. Turn on the pump and draw a vacuum of at least 380 mm Hg (15 in. Hg); note that a lower vacuum may be used, provided that it is not exceeded during the test. Determine the leakage rate. A leakage rate in excess of 4 percent of the average sampling rate or 0.00057 $m^3$/min. (0.02 cfm), whichever is less, is unacceptable.

The following leak-check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with by-pass valve fully open and coarse adjust valve completely closed. Partially open the coarse adjust valve and slowly close the by-pass valve until the desired vacuum is reached. Do not reverse direction of by-pass valve. If the desired vacuum is exceeded, either leak-check at this higher vacuum or end the leak-check as shown below and start over.

When the leak-check is completed, first slowly remove the plug from the inlet to the probe nozzle and immediately turn off the vacuum pump. This prevents water from being forced backward and keeps silica gel from being entrained backward.

4.1.4.2 Leak-Checks During Sample Run. If, during the sampling run, a component (e.g., filter assembly or impinger) change becomes necessary, a leak-check shall be conducted immediately before the change is made. The leak-check shall be done according to the procedure outlined in Section 4.1.4.1 above, except that it shall be done at a vacuum equal to or greater than the maximum value recorded up to that point in the test. If the leakage rate is found to be no greater than 0.00057 $m^3$/min (0.02 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction will need to be applied to the total volume of dry gas metered; if, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and plan to correct the sample volume as shown in Section 6.3 of this method, or shall void the sampling run.

Immediately after component changes, leak-checks are optional; if such leak-checks are done, the procedure outlined in Section 4.1.4.1 above shall be used.

4.1.4.3 Post-Test Leak-Check. A leak-check is mandatory at the conclusion of each sampling run. The leak-check shall be done in accordance with the procedures outlined in Section 4.1.4.1, except that it shall be conducted at a vacuum equal to or greater than the maximum value reached during the sampling run. If the leakage rate is found to be no greater than 0.00057 $m^3$/min (0.02 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction need be applied to the total volume of dry gas metered. If, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and correct the sample volume as shown in Section 6.3 of this method, or shall void the sampling run.

4.1.5 Particulate Train Operation. During the sampling run, maintain a sampling rate such that sampling is within 10 percent of true isokinetic, unless otherwise specified by the Administrator.

For each run, record the data required on the example data sheet shown in Figure 17-3. Be sure to record the initial dry gas meter reading. Record the dry gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made, before and after each leak check, and when sampling is halted. Take other readings required by Figure 17-3 at least once at each sample point during each time increment and additional readings when significant changes (20 percent variation in velocity head readings) necessitate additional adjustments in flow rate. Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse.

Clean the portholes prior to the test run to minimize the chance of sampling the deposited material. To begin sampling, remove the nozzle cap and verify that the pitot tube and probe extension are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs are available, which aid in the rapid adjustment to the isokinetic sampling rate without excessive computations. These nomographs are designed for use when the Type S pitot tube coefficient is 0.85±0.02, and the stack gas equivalent density (dry molecular weight) is equal to 29±4. APTD-0576 details the procedure for using the nomographs. If $C_p$ and $M_d$ are outside the above stated ranges, do not use the nomographs unless appropriate steps (see Citation 7 in Section 7) are taken to compensate for the deviations.

When the stack is under significant negative pressure (height of impinger stem), take care to close the coarse adjust valve before inserting the probe extension assembly into the stack to prevent water from being forced backward. If necessary, the pump may be turned on with the coarse adjust valve closed.

When the probe is in position, block off the openings around the probe and porthole to prevent unrepresentative dilution of the gas stream.

Traverse the stack cross section, as required by Method 1 or as specified by the Administrator, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe extension through the portholes, to minimize chance of extracting deposited material.

During the test run, take appropriate steps (e.g., adding crushed ice to the impinger ice bath) to maintain a temperature of less than 20° C (68° F) at the condenser outlet; this will prevent excessive moisture losses. Also, periodically check the level and zero of the manometer.

If the pressure drop across the filter becomes too high, making isokinetic sampling difficult to maintain, the filter may be replaced in the midst of a sample run. It is recommended that another complete filter holder assembly be used rather than attempting to change the filter itself. Before a new filter holder is installed, conduct a leak check, as outlined in Section 4.1.4.2. The total particulate weight shall include the summation of all filter assembly catches.

A single train shall be used for the entire sample run, except in cases where simultaneous sampling is required in two or more separate ducts or at two or more different locations within the same duct, or, in cases where equipment failure necessitates a change of trains. In all other situations, the use of two or more trains will be subject to the approval of the Administrator. Note that when two or more trains are used, a separate analysis of the collected particulate from each train shall be performed, unless identical nozzle sizes were used on all trains, in which case the particulate catches from the individual trains may be combined and a single analysis performed.

At the end of the sample run, turn off the pump, remove the probe extension assembly from the stack, and record the final dry gas meter reading. Perform a leak-check, as outlined in Section 4.1.4.3. Also, leak-check the pitot lines as described in Section 3.1 of Method 2; the lines must pass this leak-check, in order to validate the velocity head data.

**4.1.6 Calculation of Percent Isokinetic.** Calculate percent isokinetic (see Section 6.11) to determine whether another test run should be made. If there is difficulty in maintaining isokinetic rates due to source conditions, consult with the Administrator for possible variance on the isokinetic rates.

4.2 Sample Recovery. Proper cleanup procedure begins as soon as the probe extension assembly is removed from the stack at the end of the sampling period. Allow the assembly to cool.

When the assembly can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it to prevent losing or gaining particulate matter. Do not cap off the probe tip tightly while the sampling train is cooling down as this would create a vacuum in the filter holder, forcing condenser water backward.

Before moving the sample train to the cleanup site, disconnect the filter holder-probe nozzle assembly from the probe extension; cap the open inlet of the probe extension. Be careful not to lose any condensate, if present. Remove the umbilical cord from the condenser outlet and cap the outlet. If a flexible line is used between the first impinger (or condenser) and the probe extension, disconnect the line at the probe extension and let any condensed water or liquid drain into the impingers or condenser. Disconnect the probe extension from the condenser; cap the probe extension outlet. After wiping off the silicone grease, cap off the condenser inlet. Ground glass stoppers, plastic caps, or serum caps (whichever are appropriate) may be used to close these openings.

Transfer both the filter holder-probe nozzle assembly and the condenser to the cleanup area. This area should be clean and protected from the wind so that the chances of contaminating or losing the sample will be minimized.

Save a portion of the acetone used for cleanup as a blank. Take 200 ml of this acetone directly from the wash bottle being used and place it in a glass sample container labeled "acetone blank."

Inspect the train prior to and during disassembly and note any abnormal conditions. Treat the samples as follows:

*Container No. 1.* Carefully remove the filter from the filter holder and place it in its identified petri dish container. Use a pair of tweezers and/or clean disposable surgical gloves to handle the filter. If it is necessary to fold the filter, do so such that the particulate cake is inside the fold. Carefully transfer to the petri dish any particulate matter and/or filter fibers which adhere to the filter holder gasket, by using a dry Nylon bristle brush and/or a sharp-edged blade. Seal the container.

*Container No. 2.* Taking care to see that dust on the outside of the probe nozzle or other exterior surfaces does not get into the sample, quantitatively recover particulate matter or any condensate from the probe nozzle, fitting, and front half of the filter holder by washing these components with acetone and placing the wash in a glass container. Distilled water may be used instead of acetone when approved by the Administrator and shall be used when specified by the Administrator; in these cases, save a water blank and follow Administrator's directions on analysis. Perform the acetone rinses as follows:

Carefully remove the probe nozzle and clean the inside surface by rinsing with acetone from a wash bottle and brushing with a Nylon bristle brush. Brush until acetone rinse shows no visible particles, after which make a final rinse of the inside surface with acetone.

Brush and rinse with acetone the inside parts of the fitting in a similar way until no visible particles remain. A funnel (glass or polyethylene) may be used to aid in transferring liquid washes to the container. Rinse the brush with acetone and quantitatively collect these washings in the sample container. Between sampling runs, keep brushes clean and protected from contamination.

After ensuring that all joints are wiped clean of silicone grease (if applicable), clean the inside of the front half of the filter holder by rubbing the surfaces with a Nylon bristle brush and rinsing with acetone. Rinse each surface three times or more if needed to remove visible particulate. Make final rinse of the brush and filter holder.

Reproduced from
best available copy.

After all acetone washings and particulate matter are collected in the sample container, tighten the lid on the sample container so that acetone will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether or not leakage occurred during transport. Label the container to clearly identify its contents.

*Container No. 3.* If silica gel is used in the condenser system for mositure content determination, note the color of the gel to determine if it has been completely spent; make a notation of its condition. Transfer the silica gel back to its original container and seal. A funnel may make it easier to pour the silica gel without spilling, and a rubber policeman may be used as an aid in removing the silica gel. It is not necessary to remove the small amount of dust particles that may adhere to the walls and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, follow the procedure for Container No. 3 under "Analysis."

*Condenser Water.* Treat the condenser or impinger water as follows: make a notation of any color or film in the liquid catch. Measure the liquid volume to within ±1 ml by using a graduated cylinder or, if a balance is available, determine the liquid weight to within ±0.5 g. Record the total volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas. Discard the liquid after measuring and recording the volume or weight.

4.3 Analysis. Record the data required on the example sheet shown in Figure 17-4. Handle each sample container as follows:

*Container No. 1.* Leave the contents in the shipping container or transfer the filter and any loose particulate from the sample container to a tared glass weighing dish. Desiccate for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh to a constant weight and report the results to the nearest 0.1 mg. For purposes of this Section, 4.3, the term "constant weight" means a difference of no more than 0.5 mg or 1 percent of total weight less tare weight, whichever is greater, between two consecutive weighings, with no less than 6 hours of desiccation time between weighings.

Alternatively, the sample may be oven dried at the average stack temperature or 105° C (220° F), whichever is less, for 2 to 3 hours, cooled in the desiccator, and weighed to a constant weight, unless otherwise specified by the Administrator. The tester may also opt to oven dry the sample at the average stack temperature or 105° C (220° F), whichever is less, for 2 to 3 hours, weigh the sample and use this weight as a final weight.

*Container No. 2.* Note the level of liquid in the container and confirm on the analysis sheet whether or not leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in this container either volumetrically to ±1 ml or gravimetrically to ±0.5 g. Transfer the contents to a tared 250-ml beaker and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours and weigh to a constant weight. Report the results to the nearest 0.1 mg.

*Container No. 3.* This step may be conducted in the field. Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance.

*"Acetone Blank" Container.* Measure acetone in this container either volumetrically or gravimetrically. Transfer the acetone to a tared 250-ml beaker and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours and weigh to a constant weight. Report the results to the nearest 0.1 mg.

NOTE.—At the option of the tester, the contents of Container No. 2 as well as the acetone blank container may be evaporated at temperatures higher than ambient. If evaporation is done at an elevated temperature, the temperature must be below the boiling point of the solvent; also, to prevent "bumping," the evaporation process must be closely supervised, and the contents of the beaker must be swirled occasionally to maintain an even temperature. Use extreme care, as acetone is highly flammable and has a low flash point.

5. *Calibration.* Maintain a laboratory log of all calibrations.

5.1 Probe Nozzle. Probe nozzles shall be calibrated before their initial use in the field. Using a micrometer, measure the inside diameter of the nozzle to the nearest 0.025 mm (0.001 in.). Make three separate measurements using different diameters each time, and obtain the average of the measurements. The difference between the high and low numbers shall not exceed 0.1 mm (0.004 in.). When nozzles become nicked, dented, or corroded, they shall be reshaped, sharpened, and recalibrated before use. Each nozzle shall be permanently and uniquely identified.

5.2 Pitot Tube. If the pitot tube is placed in an interference-free arrangement with respect to the other probe assembly components, its baseline (isolated tube) coefficient shall be determined as outlined in Section 4 of Method 2. If the probe assembly is not interference-free, the pitot tube assembly coefficient shall be determined by calibration, using methods subject to the approval of the Administrator.

5.3 Metering System. Before its initial use in the field, the metering system shall be calibrated according to the procedure outlined in APTD-0576. Instead of physically adjusting the dry gas meter dial readings to correspond to the wet test meter readings, calibration factors may be used to mathematically correct the gas meter dial readings to the proper values.

Before calibrating the metering system, it is suggested that a leak-check be conducted. For metering systems having diaphragm pumps, the normal leak-check procedure will not detect leakages within the pump. For these cases the following leak-check procedure is suggested: make a 10-minute calibration run at 0.00057 $m^3$/min (0.02 cfm); at the end of the run, take the difference of the measured wet test meter and dry gas meter volumes; divide the difference by 10, to get the leak rate. The leak rate should not exceed 0.00057 $m^3$/min (0.02 cfm).

After each field use, the calibration of the metering system shall be checked by performing three calibration runs at a single, intermediate orifice setting (based on the previous field test), with the vacuum set at

Reproduced from best available copy.

the maximum value reached during the test series. To adjust the vacuum, insert a valve between the wet test meter and the inlet of the metering system. Calculate the average value of the calibration factor. If the calibration has changed by more than 5 percent, recalibrate the meter over the full range of orifice settings, as outlined in APTD-0576.

Alternative procedures, e.g., using the orifice meter coefficients, may be used, subject to the approval of the Administrator.

NOTE.—If the dry gas meter coefficient values obtained before and after a test series differ by more than 5 percent, the test series shall either be voided, or calculations for the test series shall be performed using whichever meter coefficient value (i.e., before or after) gives the lower value of total sample volume.

5.4 Temperature Gauges. Use the procedure in Section 4.3 of Method 2 to calibrate in-stack temperature gauges. Dial thermometers, such as are used for the dry gas meter and condenser outlet, shall be calibrated against mercury-in-glass thermometers.

5.5 Leak Check of Metering System Shown in Figure 17-1. That portion of the sampling train from the pump to the orifice meter should be leak checked prior to initial use and after each shipment. Leakage after the pump will result in less volume being recorded than is actually sampled. The following procedure is suggested (see Figure 17-5). Close the main valve on the meter box. Insert a one-hole rubber stopper with rubber tubing attached into the orifice exhaust pipe. Disconnect and vent the low side of the orifice manometer. Close off the low side orifice tap. Pressurize the system to 13 to 18 cm (5 to 7 in.) water column by blowing into the rubber tubing. Pinch off the tubing and observe the manometer for one minute. A loss of pressure on the manometer indicates a leak in the meter box; leaks, if present, must be corrected.

5.6 Barometer. Calibrate against a mercury barometer.

6. *Calculations.* Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after the final calculation. Other forms of the equations may be used as long as they give equivalent results.

6.1 Nomenclature.

$A_n$ = Cross-sectional area of nozzle, m² (ft²).
$B_{ws}$ = Water vapor in the gas stream, proportion by volume.
$C_a$ = Acetone blank residue concentration, mg/g.
$c_s$ = Concentration of particulate matter in stack gas, dry basis, corrected to standard conditions, g/dscm (g/dscf).
I = Percent of isokinetic sampling.
$L_a$ = Maximum acceptable leakage rate for either a pretest leak check or for a leak check following a component change; equal to 0.00057 m³/min (0.02 cfm) or 4 percent of the average sampling rate, whichever is less.
$L_i$ = Individual leakage rate observed during the leak check conducted prior to the "$i^{th}$" component change (i = 1, 2, 3 . . . n), m³/min (cfm).
$L_p$ = Leakage rate observed during the post-test leak check, m³/min (cfm).
$m_n$ = Total amount of particulate matter collected, mg.
$M_w$ = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole).
$m_a$ = Mass of residue of acetone after evaporation, mg.
$P_{bar}$ = Barometric pressure at the sampling site, mm Hg (in. Hg).
$P_s$ = Absolute stack gas pressure, mm Hg (in. Hg).
$P_{std}$ = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).
R = Ideal gas constant, 0.06236 mm Hg-m³/°K-g-mole (21.85 in. Hg-ft³/°R-lb-mole).
$T_m$ = Absolute average dry gas meter temperature (see Figure 17-3), °K (°R).
$T_s$ = Absolute average stack gas temperature (see Figure 17-3), °K (°R).
$T_{std}$ = Standard absolute temperature, 293°K (528°R).
$V_a$ = Volume of acetone blank, ml.
$V_{aw}$ = Volume of acetone used in wash, ml.
$V_{lc}$ = Total volume of liquid collected in impingers and silica gel (see Figure 17-4), ml.
$V_m$ = Volume of gas sample as measured by dry gas meter, dcm (dcf).
$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter, corrected to standard conditions, dscm (dscf).
$V_{w(std)}$ = Volume of water vapor in the gas sample, corrected to standard conditions, scm (scf).
$v_s$ = Stack gas velocity, calculated by Method 2, Equation 2-9, using data obtained from Method 17, m/sec (ft/sec).
$W_a$ = Weight of residue in acetone wash, mg.
Y = Dry gas meter calibration coefficient.
ΔH = Average pressure differential across the orifice meter (see Figure 17-3), mm $H_2O$ (in. $H_2O$).
$\rho_a$ = Density of acetone, mg/ml (see label on bottle).
$\rho_w$ = Density of water, 0.9982 g/ml (0.002201 lb/ml).
$\theta$ = Total sampling time, min.
$\theta_1$ = Sampling time interval, from the beginning of a run until the first component change, min.
$\theta_i$ = Sampling time interval, between two successive component changes, beginning with the interval between the first and second changes, min.
$\theta_p$ = Sampling time interval, from the final ($n^{th}$) component change until the end of the sampling run, min.
13.6 = Specific gravity of mercury.
60 = Sec/min.
100 = Conversion to percent.

6.2 Average dry gas meter temperature and average orifice pressure drop. See data sheet (Figure 17-3).

6.3 Dry Gas Volume. Correct the sample volume measured by the dry gas meter to standard conditions (20° C, 760 mm Hg or 68° F, 29.92 in. Hg) by using Equation 17-1.

$$V_{m(std)} = V_m Y \left(\frac{T_{std}}{T_m}\right)\left[\frac{P_{bar} + \frac{\Delta H}{13.6}}{P_{std}}\right]$$

$$= K_1 V_m Y \frac{P_{bar} + (\Delta H/13.6)}{T_m}$$

Equation 17-1

where:

$K_1$ = 0.3858° K/mm Hg for metric units; 17.64° R/in. Hg for English units.

NOTE.—Equation 17-1 can be used as written unless the leakage rate observed during any of the mandatory leak checks (i.e., the post-test leak check or leak checks conducted prior to component changes) exceeds $L_a$. If $L_p$ or $L_i$ exceeds $L_a$, Equation 17-1 must be modified as follows:

(a) Case I. No component changes made during sampling run. In this case, replace $V_m$ in Equation 17-1 with the expression:

$$[V_m - (L_p - L_a)\theta]$$

(b) Case II. One or more component changes made during the sampling run. In this case, replace $V_m$ in Equation 17-1 by the expression:

$$[V_m - (L_1 - L_a)\,\theta_1 - \sum_{i=2}^{n} (L_i - L_a)\,\theta_i - (L_p - L_a)\,\theta_p]$$

and substitute only for those leakage rates ($L_i$ or $L_p$) which exceed $L_a$.

6.4 Volume of water vapor.

$$V_{w(std)} = V_{1c}\left(\frac{\rho_w}{M_w}\right)\left(\frac{RT_{std}}{P_{std}}\right) = K_2\,V_{1c}$$

Equation 17-2

where:

$K_2$ = 0.001333 m³/ml for metric units; 0.04707 ft³/ml for English units.

6.5 Moisture Content.

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}}$$

Equation 17-3

6.6 Acetone Blank Concentration.

$$C_a = \frac{m_a}{V_a\,\rho_a}$$

Equation 17-4

6.7 Acetone Wash Blank.

$$W_a = C_a V_{aw} \rho_a$$

Equation 17-5

6.8 Total Particulate Weight. Determine the total particulate catch from the sum of the weights obtained from containers 1 and 2 less the acetone blank (see Figure 17-4).

NOTE.—Refer to Section 4.1.5 to assist in calculation of results involving two or more filter assemblies or two or more sampling trains.

6.9 Particulate Concentration.

$$c_s = (0.001\ g/mg)\,(m_n/V_{m(std)})$$

Equation 17-6

6.10 Conversion Factors:

| From | To | Multiply by |
|---|---|---|
| scf | m³ | 0.02832 |
| g/ft³ | gr/ft³ | 15.43 |
| g/ft³ | lb/ft³ | $2.205 \times 10^{-3}$ |
| g/ft³ | g/m³ | 35.31 |

6.11 Isokinetic Variation.

6.11.1 Calculation from Raw Data.

$$I = \frac{100\,T_s\,[K_3 V_{1c} + (V_m Y/T_m)\,(P_{bar} + \Delta H/13.6)]}{60\,\theta v_s\,P_s\,A_n}$$

Equation 17-7

where:

$K_3$ = 0.003454 mm Hg-m³/ml-°K for metric units; 0.002669 in. Hg-ft³/ml-°R for English units.

6.11.2 Calculation from Intermediate Values.

$$I = \frac{T_s\,V_{m(std)}\,P_{std}\,100}{T_{std}\,v_s\,\theta\,A_n\,P_s\,60\,(1-B_{ws})}$$

$$= K_4\,\frac{T_s\,V_{m(std)}}{P_s\,v_s\,A_n\,\theta\,(1-B_{ws})}$$

Equation 17-8

where:

$K_4$ = 4.320 for metric units; 0.09450 for English units.

6.12 Acceptable Results. If 90 percent 010110 percent, the results are acceptable. If the results are low in comparison to the standard and I is beyond the acceptable range, or, if I is less than 90 percent, the Administrator may opt to accept the results. Use Citation 4 in Section 7 to make judgments. Otherwise, reject the results and repeat the test.

7. *Bibliography.*

1. Addendum to Specifications for Incinerator Testing at Federal Facilities. PHS, NCAPC. December 6, 1967.

2. Martin, Robert M., Construction Details of Isokinetic Source Sampling Equipment. Environmental Protection Agency. Research Triangle Park, N.C. APTD-0581. April, 1971.

3. Rom, Jerome J., Maintenance, Calibration, and Operation of Isokinetic Source-Sampling Equipment. Environmental Protection Agency. Research Triangle Park, N.C. APTD-0576. March, 1972.

4. Smith, W. S., R. T. Shigehara, and W. F. Todd. A Method of Interpreting Stack Sampling Data. Paper Presented at the 63rd Annual Meeting of the Air Pollution Control Association, St. Louis, Mo. June 14-19, 1970.

5. Smith, W. S., et al., Stack Gas Sampling Improved and Simplified with New Equipment. APCA Paper No. 67-119. 1967.

6. Specifications for Incinerator Testing at Federal Facilities. PHS, NCAPC. 1967.

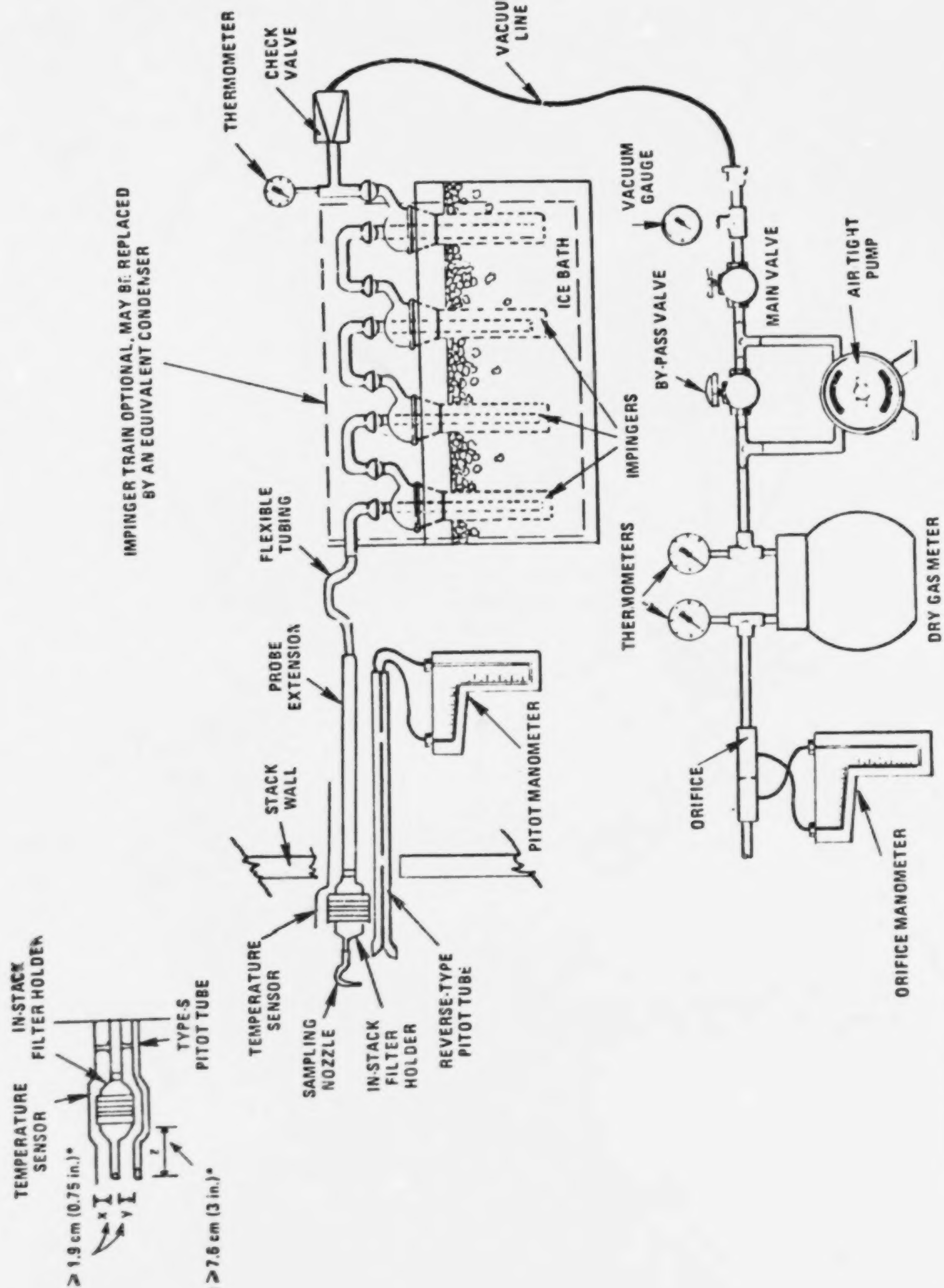
7. Shigehara, R. T., Adjustments in the EPA Nomograph for Different Pitot Tube Coefficients and Dry Molecular Weights. Stack Sampling News 2:4-11. October, 1974.

8. Vollaro, R. F., A Survey of Commercially Available Instrumentation for the Measurement of Low-Range Gas Velocities. U.S. Environmental Protection Agency, Emission Measurement Branch. Research Triangle Park, N.C. November, 1976 (unpublished paper).

9. Annual Book of ASTM Standards. Part 26. Gaseous Fuels; Coal and Coke; Atmo-

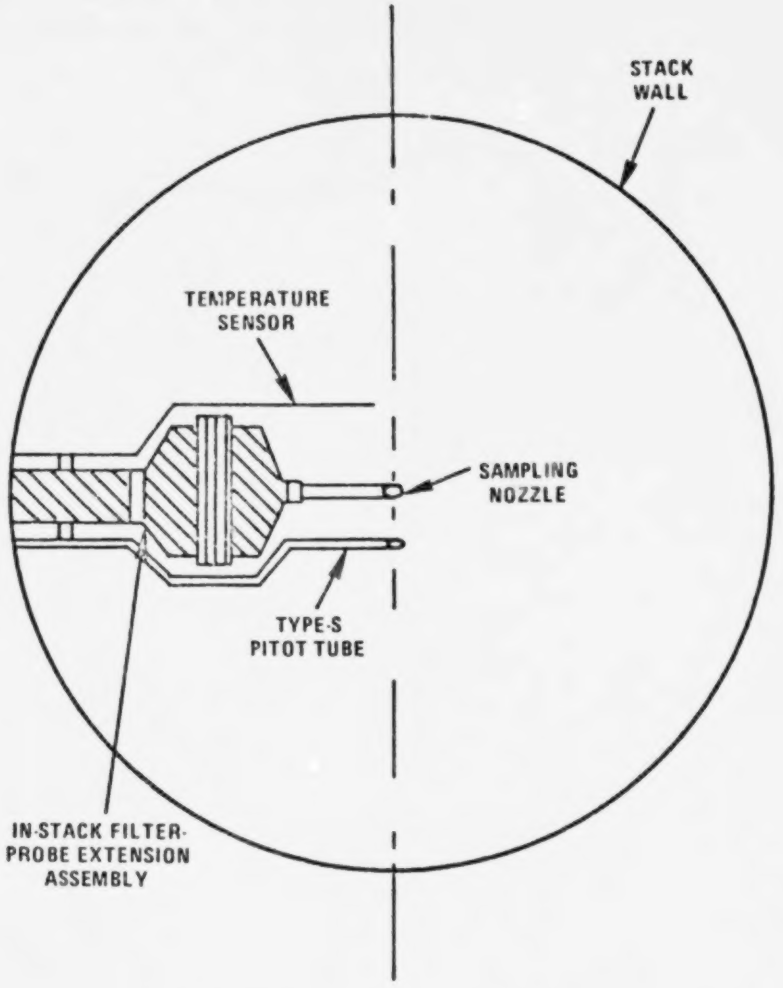
spheric Analysis. American Society for Testing and Materials. Philadelphia, Pa. 1974. pp. 617-622.

10. Vollaro, R. F., Recommended Procedure for Sample Traverses in Ducts Smaller than 12 Inches in Diameter. U.S. Environmental Protection Agency, Emission Measurement Branch. Research Triangle Park, N.C. November, 1976


IMPINGER TRAIN OPTIONAL, MAY BE REPLACED BY AN EQUIVALENT CONDENSER
THERMOMETER
CHECK VALVE
VACUUM LINE
FLEXIBLE TUBING
ICE BATH
IMPINGERS
VACUUM GAUGE
MAIN VALVE
AIR TIGHT PUMP
BY-PASS VALVE
THERMOMETERS
DRY GAS METER
PROBE EXTENSION
PITOT MANOMETER
STACK WALL
ORIFICE
ORIFICE MANOMETER
TEMPERATURE SENSOR
SAMPLING NOZZLE
IN-STACK FILTER HOLDER
REVERSE-TYPE PITOT TUBE
TEMPERATURE SENSOR
IN-STACK FILTER HOLDER
TYPE-S PITOT TUBE
x
y
z
x = y ≥ 1.9 cm (0.75 in.)*
z ≥ 7.6 cm (3 in.)*

*SUGGESTED (INTERFERENCE-FREE) SPACINGS

Figure 17-1. Particulate-Sampling Train, Equipped with In-Stack Filter.


STACK WALL
TEMPERATURE SENSOR
SAMPLING NOZZLE
TYPE-S PITOT TUBE
IN-STACK FILTER-PROBE EXTENSION ASSEMBLY

$$\text{ESTIMATED BLOCKAGE (\%)} = \left[\frac{\text{SHADED AREA}}{\text{DUCT AREA}}\right] \times 100$$

Figure 17-2. Projected-area model of cross-section blockage (approximate average for a sample traverse) caused by an in-stack filter holder-probe extension assembly.

PLANT ____________

LOCATION ____________

OPERATOR ____________

DATE ____________

RUN NO. ____________

SAMPLE BOX NO. ____________

METER BOX NO. ____________

METER ΔH@ ____________

C FACTOR ____________

PITOT TUBE COEFFICIENT, Cp ____________

SCHEMATIC OF STACK CROSS SECTION

BAROMETRIC PRESSURE ____________

ASSUMED MOISTURE, % ____________

PROBE EXTENSION LENGTH, m(ft.) ____________

NOZZLE IDENTIFICATION NO. ____________

AVERAGE CALIBRATED NOZZLE DIAMETER cm(in.) ____________

FILTER NO. ____________

LEAK RATE, $m^3$/min,(cfm) ____________

STATIC PRESSURE, mm Hg (in. Hg) ____________

| TRAVERSE POINT NUMBER | SAMPLING TIME (θ), min. | VACUUM mm Hg (in. Hg) | STACK TEMPERATURE ($T_S$), °C (°F) | VELOCITY HEAD ($\Delta P_S$), mm $H_2O$ (in. $H_2O$) | PRESSURE DIFFERENTIAL ACROSS ORIFICE METER, mm $H_2O$ (in. $H_2O$) | GAS SAMPLE VOLUME, $m^3$ ($ft^3$) | GAS SAMPLE TEMPERATURE AT DRY GAS METER | | TEMPERATURE OF GAS LEAVING CONDENSER OR LAST IMPINGER, °C (°F) |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | INLET, °C (°F) | OUTLET, °C (°F) | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| TOTAL | | | | | | | Avg | Avg | |
| AVERAGE | | | | | | | Avg | | |

Figure 17-3. Particulate field data.

Plant ____________________________________

Date ____________________________________

Run No. ____________________________________

Filter No. ____________________________________

Amount liquid lost during transport ____________________

Acetone blank volume, ml __________________________

Acetone wash volume, ml __________________________

Acetone black concentration, mg/mg (equation 17-4) ____________

Acetone wash blank, mg (equation 17-5) ________________

| CONTAINER NUMBER | WEIGHT OF PARTICULATE COLLECTED, mg | | |
|---|---|---|---|
| | FINAL WEIGHT | TARE WEIGHT | WEIGHT GAIN |
| 1 | | | |
| 2 | | | |
| TOTAL | | | |
| | | Less acetone blank | |
| | | Weight of particulate matter | |

| | VOLUME OF LIQUID WATER COLLECTED | | |
|---|---|---|---|
| | IMPINGER VOLUME, ml | SILICA GEL WEIGHT, g | |
| FINAL | | | |
| INITIAL | | | |
| LIQUID COLLECTED | | | |
| TOTAL VOLUME COLLECTED | | g* | ml |

* CONVERT WEIGHT OF WATER TO VOLUME BY DIVIDING TOTAL WEIGHT INCREASE BY DENSITY OF WATER (1g/ml).

$$\frac{\text{INCREASE, g}}{1\ \text{g/ml}} = \text{VOLUME WATER, ml}$$

Figure 17-4. Analytical data.

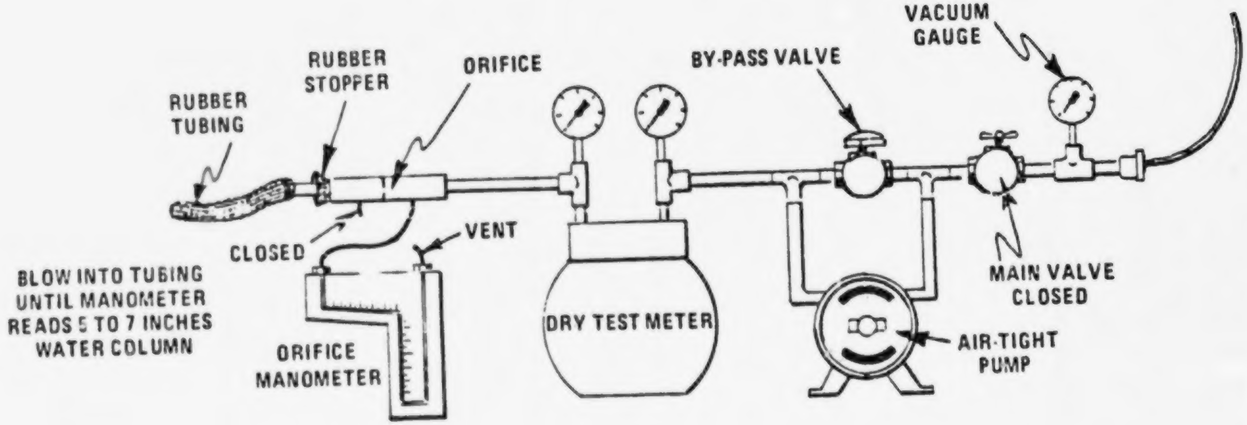

RUBBER TUBING
RUBBER STOPPER
ORIFICE
BY-PASS VALVE
VACUUM GAUGE
CLOSED
VENT
BLOW INTO TUBING UNTIL MANOMETER READS 5 TO 7 INCHES WATER COLUMN
ORIFICE MANOMETER
DRY TEST METER
MAIN VALVE CLOSED
AIR-TIGHT PUMP

Figure 17-5. Leak check of meter box.

# APPENDIX F

## FEDERAL STATIONARY SOURCE PERFORMANCE STANDARDS

Since December 1971 the Environmental Protection Agency has promulgated source performance standards for twenty industrial air pollution sources. These regulations have been presented in numerous issues of the Federal Register and can now be found in the Code of Federal Regulations under Title 40 - Protection of Environment; Chapter 1 - Environmental Protection Agency; Subchapter C - Air Programs; Part 60 - Standards of Performance For New Stationary Sources.

This appendix presents a summary of these regulations as found in the Code of Federal Regulations. This summary is an updated listing as found originally in the following reference:

L.S. Chaput. 1976. Federal Standards of Performance For New Stationary Sources Of Air Pollution - A Summary of Regulations. Journal of the Air Pollution Control Association. Vol. 26, No. 11; 1055-1060.

# Standards of Performance – 40 CFR Part 60.

| Source category | Affected facility | Pollutant | Emission level | Monitoring requirement |
|---|---|---|---|---|
| **Subpart D:**<br>**Steam generators**<br>(>250 million Btu/hr)<br>*Promulgated*<br>12/23/71 (36 FR 24876)<br>*Revised*<br>7/26/72 (37 FR 14877)<br>6/14/74 (39 FR 20790)<br>1/16/75 (40 FR 2803)<br>10/6/75 (40 FR 46250)<br>3/7/78(43FR9278) | Coal fired boilers | Particulate | 0.10 lb/$10^6$ Btu | No requirement |
| | | Opacity | 20% | Continuous |
| | | $SO_2$ | 1.2 lb/$10^6$ Btu | Continuous |
| | | $NO_x$ (except lignite and coal refuse) | 0.70 lb/$10^6$ Btu | Continuous |
| | Oil fired boilers | Particulate | 0.10 lb/$10^6$ Btu | No requirement |
| | | Opacity | 20% ; 40% 2 min/hr | Continuous |
| | | $SO_2$ | 0.80 lb/$10^6$ Btu | Continuous |
| | | $NO_x$ | 0.30 lb/$10^6$ Btu | Continuous |
| | Gas fired boilers | Particulate | 0.10 lb/$10^6$ Btu | No requirement |
| | | Opacity | 20% | No requirement |
| | | $NO_x$ | 0.20 lb/$10^6$ Btu | Continuous |
| **Subpart E:**<br>**Incinerators**<br>(>50 tons/day)<br>*Promulgated*<br>12/23/71 (36 FR 24876)<br>*Revised*<br>6/14/74 (39 FR 20790) | Incinerators | Particulate | 0.80 gr/dscf corrected to 12% CO | No requirement |
| **Subpart F:**<br>**Portland cement plants**<br>*Promulgated*<br>12/23/71 (36 FR 24876)<br>*Revised*<br>6/14/74 (39 FR 20790)<br>11/12/74 (39 FR 39874)<br>10/6/75 (40 FR 46250) | Kiln | Particulate | 0.30 lb/ton | No requirement |
| | | Opacity | 20% | No requirement |
| | Clinker cooler | Particulate | 0.10 lb/ton | No requirement |
| | | Opacity | 10% | No requirement |
| | Fugitive Emission points | Opacity | 10% | No requirement |
| **Subpart G:**<br>**Nitric acid plants**<br>*Promulgated*<br>12/23/71 (36 FR 24876)<br>*Revised*<br>5/23/73 (38 FR 13562)<br>6/14/74 (39 FR 20790)<br>10/6/75 (40 FR 46250) | Process equipment | Opacity | 10% | No requirement |
| | | $NO_x$ | 3.0 lb/ton | Continuous |
| **Subpart H:**<br>**Sulfuric acid plants**<br>*Promulgated*<br>12/23/71 (36 FR 24876)<br>*Revised*<br>5/23/73 (38 FR 13562)<br>6/14/74 (39 FR 20790)<br>10/6/75 (40 FR 46250) | Process equipment | $SO_2$ | 4.0 lb/ton | Continuous |
| | | Acid mist | 0.15 lb/ton | No requirement |
| | | Opacity | 10% | No requirement |

| Source category | Affected facility | Pollutant | Emission level | Monitoring requirement |
|---|---|---|---|---|
| **Subpart I:**<br>**Asphalt concrete plants**<br>*Promulgated*<br>3/8/74 (39 FR 9308)<br>*Revised*<br>10/6/75 (40 FR 46250) | Dryers; screening and weighing systems; storage, transfer, and loading systems; and dust handling equipment | Particulate | 0.04 gr/dscf<br>(90 mg/dscm) | No requirement |
| | | Opacity | 20% | No requirement |
| **Subpart J:**<br>**Petroleum refineries**<br>*Promulgated*<br>3/8/74 (39 FR 9308)<br>*Revised*<br>10/6/75 (40 FR 46250)<br>3/15/78(43FR10868) | Catalytic cracker | Particulate | 1.0 lb/1000 lb | No requirement |
| | | Opacity | 30% (3 min. exemption) | Continuous |
| | | CO | 0.05% | Continuous |
| | Fuel gas combination | $SO_2$ | 0.1 gr $H_2S$/dscf<br>(230 mg/dscm) | Continuous |
| **Subpart K:**<br>**Storage vessels for petroleum liquids**<br>*Promulgated*<br>3/8/74 (39 FR 9308)<br>*Revised*<br>4/17/74 (39 FR 13776)<br>6/14/74 (39 FR 20790) | Storage tanks >40,000 gal. capacity | Hydrocarbons | For vapor pressure 78-570 mm Hg, equip with floating roof, vapor recovery system, or equivalent; for vapor pressure >570 mm Hg, equip with vapor recovery system or equivalent | No requirement |
| **Subpart L:**<br>**Secondary lead smelters**<br>*Promulgated*<br>3/8/74 (39 FR 9308)<br>*Revised*<br>4/17/74 (39 FR 13776)<br>10/6/75 (40 FR 46250) | Reverberatory and blast furnaces | Particulate | 0.022 gr/dscf<br>(50 mg/dscm) | No requirement |
| | | Opacity | 20% | No requirement |
| | Pot furnaces | Opacity | 10% | No requirement |
| **Subpart M:**<br>**Secondary brass and bronze plants**<br>*Promulgated*<br>3/8/74 (39 FR 9308)<br>*Revised*<br>10/6/75 (40 FR 46250) | Reverberatory furnace | Particulate | 0.022 gr/dscf<br>(50 mg/dscm) | No requirement |
| | | Opacity | 20% | No requirement |
| | Blast and electric furnaces | Opacity | 10% | No requirement |
| **Subpart N:**<br>**Iron and steel plants**<br>*Promulgated*<br>3/8/74 (39 FR 9308) | Basic oxygen process furnace | Particulate | 0.022 gr/dscf<br>(50 mg/dscm) | No requirement |

| Source category | Affected facility | Pollutant | Emission level | Monitoring requirement |
|---|---|---|---|---|
| **Subpart O:** | | | | |
| **Sewage treatment plants** | Sludge incinerators | Particulate | 1.30 lb/ton | Mass or volume of sludge |
| | | Opacity | 20% | No requirement |
| *Promulgated*<br>3/8/74 (39 FR 9308) | | | | |
| *Revised*<br>4/17/74 (39 FR 13776)<br>5/3/74 (39 FR 15396)<br>10/6/75 (40 FR 46250) | | | | |
| **Subpart P:** | | | | |
| **Primary copper smelters** | Dryer | Particulate | 0.022 gr/dscf (50 mg/dscm) | No requirement |
| | | Opacity | 20% | Continuous |
| *Promulgated*<br>1/15/76 (41 FR 2331) | Roaster, smelting furnace,* copper converter | $SO_2$ | 0.065% | Continuous |
| *Revised*<br>2/26/76 (41 FR 8346) | | Opacity | 20% | No requirement |
| | *Reverberatory furnaces that process high-impurity feed materials are exempt from $SO_2$ standard | | | |
| **Subpart Q:** | | | | |
| **Primary zinc smelters** | Sintering machine | Particulate | 0.022 gr/dscf (50 mg/dscm) | No requirement |
| | | Opacity | 20% | Continuous |
| *Promulgated*<br>1/15/76 (41 FR 2331) | Roaster | $SO_2$ | 0.065% | Continuous |
| | | Opacity | 20% | No requirement |
| **Subpart R.:** | | | | |
| **Primary lead smelters** | Blast or reverberatory furnace, sintering macnine discharge end | Particulate | 0.022 gr/dscf (50 mg/dscm) | No requirement |
| | | Opacity | 20% | Continuous |
| *Promulgated*<br>1/15/76 (41 FR 2331) | Sintering machine, electric smelting furnace, converter | $SO_2$ | 0.065% | Continuous |
| | | Opacity | 20% | No requirement |
| **Subpart S:** | | | | |
| **Primary aluminum reduction plants** | Potroom group<br>(a) Soderberg plant | (a) Total fluorides | 2.0 lb/ton | No requirement |
| | | Opacity | 10% | No requirement |
| *Promulgated*<br>1/26/76 (41 FR 3825) | (b) Prebake plant | (b) Total fluorides | 1.9 lb/ton | No requirement |
| | | Opacity | 10% | No requirement |
| | Anode bake plants | Total fluorides | 0.1 lb/ton | No requirement |
| | | Opacity | 20% | No requirement |

| Source category | Affected facility | Pollutant | Emission level | Monitoring requirement |
|---|---|---|---|---|
| **Subpart T:**<br>**Phosphate fertilizer plants**<br>*Promulgated*<br>8/6/75 (40 FR 33152) | Wet process phosphoric acid | Total fluorides | 0.02 lb/ton | Total pressure drop across process scrubbing system |
| **Subpart U:** | Superphosphoric acid | Total fluorides | 0.01 lb/ton | Total pressure drop across process scrubbing system |
| **Subpart V:** | Diammonium phosphate | Total fluorides | 0.06 lb/ton | Total pressure drop across process scrubbing system |
| **Subpart W:** | Triple super-phosphate | Total fluorides | 0.2 lb/ton | Total pressure drop across process scrubbing system |
| **Subpart X:** | Granular triple superphosphate | Total fluorides | $5.0 \times 10^{-4}$ lb/hr/ton | Total pressure drop across process scrubbing system |
| **Subpart Y:**<br>**Coal preparation plants**<br>*Promulgated*<br>1/15/76 (41 FR 2232) | Thermal dryer | Particulate | 0.031 gr/dscf<br>(0.070 g/dscm) | Temperature<br>Scrubber pressure loss<br>Water pressure |
| | | Opacity | 20% | No requirement |
| | Pneumatic coal cleaning equipment | Particulate | 0.018 gr/dscf<br>(0.040 g/dscm) | No requirement |
| | | Opacity | 10% | No requirement |
| | Processing and Conveying equipment, storage systems, transfer and loading systems | Opacity | 20% | No requirement |
| **Subpart Z:**<br>**Ferroalloy production facilities**<br>*Promulgated*<br>5/4/76 (41 FR 18497)<br>*Revised*<br>5/20/76 (41 FR 20659) | Electric submerged arc furnaces | Particulate | 0.99 lb/Mw-hr<br>(0.45 kg/Mw-hr)<br>("high silicon alloys")<br>0.51 lb/Mw-hr<br>(0.23 kg/Mw-hr)<br>(chrome and manganese alloys) | No requirement |
| | | | No visible emissions may escape furnace capture system | Flowrate monitoring in hood |

| Source category | Affected facility | Pollutant | Emission level | Monitoring requirement |
|---|---|---|---|---|
| **Ferroalloy production facilities (cont.)** | | | No visible emission may escape tapping system for >40% of each tapping period | Flowrate monitoring in hood |
| | | Opacity | 15% | Continuous |
| | | CO | 20% volume basis | No requirement |
| | Dust handling equipment | Opacity | 10% | No requirement |
| **Subpart AA:**<br>**Iron and steel plants**<br>*Promulgated* 9/23/75 (40 FR 43850) | Electric arc furnaces | Particulate | 0.0052 gr/dscf (12 mg/dscm) | No requirement |
| | | Opacity<br>(a) control device | 3% | Continuous |
| | | (b) shop roof | 0, except<br>20%—charging<br>40%—tapping | Flowrate monitoring in capture hood<br>Pressure monitoring in DSE system |
| | Dust handling equipment | Opacity | 10% | No requirement |
| **Subpart BB:**<br>**Kraft pulp mills**<br>*Promulgated* 2/23/78(43FR7572) | Straight and cross recovery furnace | Particulate | 0.044 gr/dscf corrected to 8% oxygen | |
| | | Opacity | 35% | Continuous |
| | Straight kraft recovery furnace | Total reduced sulfur | 5 ppm by volume on a dry basis, corrected to 8% oxygen | Continuous |
| | Cross recovery furnace | Total reduced sulfur | 25 ppm by volume on a dry basis, corrected to 8% oxygen | Continuous |
| | Smelt dissolving tank | Particulate | 0.1 g/kg black liquor solids (dry weight) | |
| | | Total reduced sulfur | 0.0084 g/kg black liquor solids (dry weight) | Continuous |
| | Lime kiln | Particulate | 0.067 gr/dscf corrected to 10 % oxygen when gaseous fossil fuel is burned<br>0.13 gr/dscf corrected to 10% oxygen when liquid fossil fuel is burned | |
| | | Total reduced sulfur | 8 ppm by volume on a dry basis, corrected to 10% oxygen | Continuous |
| | Digester system, brown stock washer system, multiple-effect evaporator system, black liquor oxidation system, condensate stripper system | Total reduced sulfur | 5 ppm by volume on a dry basis, corrected to 10% oxygen, except as noted in CFR. | Continuous |

# BIBLIOGRAPHY

Air Pollution Control Association (ed.). Proceedings of a Specialty Conference on: The User and Fabric Filtration Equipment. October 14-16, 1973, Buffalo, New York. 176 pp.

Air Pollution Control Association (ed.). Proceedings of a Second Specialty Conference on: The User and Fabric Filtration Equipment. October 5-7, 1975, Buffalo, New York. 197 pp.

Allen, T. 1975. Particle Size Measurement. John Wiley and Sons, New York, New York. 454 pp.

American Air Filter Company. Proceedings: 2nd International Fabric Alternatives Forum, Denver, Colorado, July 27 and 28, 1977.

American Society For Testing and Materials. Standard Method For Sampling Stacks For Particulate Matter. Designation D2928-71, Annual Book of ASTM Standards, 1977.

American Society of Mechanical Engineers. Power Test Code-27: Determining Dust Concentrations in a Gas Stream. ASME, New York, April, 1957.

Batel, W. State of Development and Tendencies for the Filtering Dust Collector. Staub Reinhaltung der Luft in English, 33 (1973), No. 9, September. pp. 352-362.

## Bibliography (Cont'd.)

Billings, C. E. (ed.). Proceedings: Fabric Filter Symposium March 16-18, 1971, Charleston, South Carolina. Sponsored by Division of Process Control Engineering, Air Pollution Control Office, U.S. Environmental Protection Agency.

Billings, C. E., and J. Wilder. Handbook of Fabric Filter Technology. Vol. I, II, III, IV. Furnished to the National Air Pollution Control Administration Under Contract Number CPA-22-69-33 by GCA Corporation. December 1970.

Bird, A. N., Jr. 1973. Summary of Techniques Used for Measuring Concentration and Size of Fine Particulate Emission. Memorandum Prepared for the Working Group for Stationary Source Air Pollution Control Technology. Southern Research Institute, Birmingham, Alabama. 14 pp.

Blake, D. E. 1976. Symposium on Particulate Control in Energy Processes. EPA-600/7-76-010. U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711. 584 pp.

Blake, D. E. Source Assessment Sampling System: Design and Development. EPA-600/7-78-018, February, 1978. U.S. Environmental Protection Agency, Research Triangle Park, NC. 221 pp.

Bradway, R. M., and R. W. Cass. Fractional Efficiency of a Utility Boiler Baghouse - Nucla Generating Plant. EPA-600/2-75-013a, U.S. Environmental Protection Agency, Washington, D.C., August, 1975. 148 pp.

## Bibliography (Cont'd.)

Brenchley, D. L., G. D. Turley, and R. F. Yarmac. 1974. Industrial Source Sampling. Ann Arbor Science Publishers, Inc., Ann Arbor, Michigan. 484 pp.

Breslin, A. J., S. F. Guggenheim, and A. C. George. Compact High Efficiency Diffusion Batteries. Staub (in English), 31(8), pp. 1-5 (1971).

Cass, R. W., and R. M. Bradway. Fractional Efficiency of a Utility Boiler Baghouse: Sunbury Steam-Electric Station. EPA-600/2-76-077a, U.S. Environmental Protection Agency, Washington, D.C., March, 1976. 244 pp.

Chang, Hsing-Chi. A Parallel Multicyclone Size-Selective Particulate Sampling Train. Amer. Ind. Hyg. Assoc. J., September, 1974.

Cooper, Douglas W., and Vladimir Hampl. Fabric Filtration Performance Model. In: Proc. Conf. Particulate Collection Problems Converting Low Sulfur Coals. EPA-600/7-76-016, U.S. Environmental Protection Agency, Research Triangle Park, NC, 1976. pp. 149-185.

Cushing, K. M., G. Lacey, J. D. McCain, and W. B. Smith. Particulate Sizing Techniques for Control Device Evaluation: Cascade Impactor Calibrations. EPA-600/2-76-280, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, October, 1976, 94 pp, NTIS-PB-245184 ($5.75).

Davies, C. N. 1966. Aerosol Science. Academic Press, New York, New York 10003. 468 pp.

Davies, C. N. and M. Aylward. Proc. Phys. Soc., (64):889, 1951.

Dennis, Richard, and J. Wilder. Fabric Filter Cleaning Studies. EPA-650/2-75-009, U.S. Environmental Protection Agency, Washington, D.C., January, 1975. 438 pp.

Dennis, R., R. W. Cass, and J. E. Langley. Development of Data Base and Fabric Filtration Models for Design of Particulate Control Systems, Field Related Tests. Contract No. 68-02-1438, U.S. Environmental Protection Agency, Research Triangle Park, NC, 1975. 119 pp.

Dorman, R. G. Chapter VIII. In: Aerosol Science, C. N. Davies, ed. New York, Academic Press, 1966.

Draemel, D. C. Relationship Between Fabric Structure and Filtration Performance in Dust Filtration. EPA-R2-73-288, U. S. Environmental Protection Agency, Research Triangle Park, North Carolina, July, 1973. 83 pp.

Englund, H. M., and W. T. Berry (ed.). Control Technology: Particulates. APCA Reprint Series. Air Pollution Control Association, Pittsburgh, PA, July, 1973. 80 pp.

Ensor, David S., R. G. Hooper, and R. W. Scheck. Determination of the Fractional Efficiency, Opacity Characteristics, Engineering and Economic Aspects of a Fabric Filter Operating on a Utility Boiler. Electric Power Research Institute Report No. EPRI FP-297. November, 1976.

## Bibliography (Cont'd.)

Ensor, D. S. and B. S. Jackson. Evaluation of a Particulate Scrubber On A Coal-Fired Utility Boiler. EPA-600/2-75-074, 1975. U.S. Environmental Protection Agency, Research Triangle Park, NC 27711.

Federal Register. Standards of Performance For New Stationary Sources: Revision to Reference Methods 1-8. Volume 42, Number 160, Thursday, August 18, 1977, pp. 41753-41789.

Federal Register. Standards of Performance for New Stationary Sources. Vol. 36, No. 247, December 23, 1971.

Felix, L. G., G. I. Clinard, G. E. Lacey, and J. D. McCain. Inertial Cascade Impactor Substrate Media For Flue Gas Sampling. Interagency Energy-Environment Research and Development Program Report, EPA-600/7-77-060, June, 1977.

Fuchs, N. A., I. B. Stechkina, and V.I. Starasselskii. On The Determination Of Particle Size Distribution On Polydisperse Aerosols By The Diffusion Method. Brit. J. Appl. Phys., 13, 280-81 (1962).

Fuchs, N. A. The Mechanics of Aerosols. The MacMillan Co. 60 - 5th Avenue, NY (1954), pp. 204-12.

Haberl, J. B. and S. J. Fusco. Condensation Nuclei Counters: Theory and Principles of Operation. General Electric Technical Information Series, No. 70-POD 12 (1970).

Hall, R. R., and R. Dennis. Mobile Fabric Filter System Design and Field Test Results. EPA-650/2-75-059, U.S. Environmental Protection Agency, Washington, D.C., July, 1975. 136 pp.

Hamersma, J. W., S. L. Reynolds, and R. F. Maddalone. Procedures Manual for Level 1 Environmental Assessment. EPA-600/2-76-160a, EPA, Research Triangle Park, North Carolina.

Harris, D. B. Procedures For Cascade Impactor Calibration And Operation In Process Streams. U.S. Environmental Protection Agency Report No. EPA-600/2-77-004, January, 1977.

Hewitt, G. W. The Charging of Small Particles for Electrostatic Precipitation. AIEE Winter General Meeting, New York. Paper No. 73-283, 1957.

Holland, C. R., and E. Rothwell. Model Studies of Fabric Dust Filtration 1. Flow Characteristics of Dust Cakes Uniformly Distributed on Filter Fabrics. Filtration Separation 14:30, 1977.

Huang, C. M., B. G. McKinney, G. A. Hollinden, and J. R. Crooks. Review of Major Developments In Particulate Control Technologies in USA. Tennessee Valley Authority, Chattanooga, Tennessee, October, 1976.

Johnson, J. W., G. I. Clinard, L. G.Felix, and J. D. McCain. A Computer-Based Cascade Impactor Data Reduction System. EPA-600/7-78-042, March, 1978. U.S. Environmental Protection Agency, Research Triangle Park, N.C. 602 pp.

Jones, A. A. Theory and Application of Fabric Dust Filters. Pit and Quarry, 9 (n.a.), 110-120, 1972.

Koscianowski, J. R. and L. Koscianowski. Effect of Filtration Parameters on Dust Cleaning Fabrics. EPA-600/2-76-074, U.S. Environmental Protection Agency, Washington, D.C., March, 1976. 185 pp.

Koscianowski, L., J. Zareba, and J. Koscianowski. Study of Fabric Filters For Industrial Dust Collecting Devices. National Technical Information Service Report No. 258835-T, 1974.

Limbach, R. F. Nodular Deposits in Fabric Filters. Paper No. 77-51.6. Presented at the 70th Annual Meeting of the Air Pollution Control Association, Toronto, Canada. June 20-24, 1977.

Liu, B. Y. H., and D. Y. H. Pui. A Sub-micron Aerosol Standard And The Primary, Absolute Calibration Of The Condensation Nuclei Counter. J. of Colloid and Interface Science, 47(1), (1974).

Liu, B. Y. H., and D. Y. H. Pui. On the Performance of the Electrical Aerosol Analyzer. J. Aerosol Science, 6, pp. 249-64 (1975).

Liu, B. Y. H. (ed.). 1976. Fine Particles: Aerosol Generation, Measurement, Sampling, and Analysis. Academic Press. New York, New York. 837 pp.

Liu, B. Y. H., K. T. Whitby, and D. Y. H. Pui. A Portable Electrical Aerosol Analyzer for Size Distribution Measurements of Submicron Aerosols. APCA J. 24(11):1067-72, 1974.

## Bibliography (Cont'd)

MacLauren, E., and C. Junge. Relationship of Cloud Nuclei-System to Aerosol Size Distribution and Composition. J. of Atmospheric Science, 4, (1971).

Maddalone, R. and N. Garner. Process Measurements Procedures - Sulfuric Acid Emissions. TRW Systems Group Document 28055-6004-R0-00, February, 1977.

Marlow, W. H., P. C. Reist, and G. A. Dwiggins. Aspects of the Performance of the Electrical Aerosol Analyses Under Non Ideal Conditions, J. Aerosol Science, 7, 457 (1976).

Marple, V. A. and K. L. Rubow. Aerodynamic Particle Size Calibrations of Optical Particle Counters. J. Aerosol Science, 7, pp. 425-438 (1976).

Marple, V. A. and K. Willeke. 1976. Impactor Design. Atmospheric Environment. Vol 10: 891-896.

Marple, V. A. Doctoral Thesis, University of Minnesota, Minneapolis, MN, 1970.

McCain, J. D., K. M. Cushing, and A. N. Bird, Jr. 1973. Field Measurements Of Particle Size Distribution With Inertial Sizing Devices. EPA-650/2-73-035. U.S. Environmental Protection Agency, Washington, D.C. 20460. 52 pp. NTIS-PB 226292/$.75.

McCain, J. D., K. M. Cushing, and W. B. Smith. 1974. Methods For Determining Particulate Mass and Size Properties: Laboratory and Field Measurements. Journal of the Air Pollution Control Association, Vol. 24, No. 12: 1172-1176.

# Bibliography (Cont'd.)

McKee, D. E., R. E. Lucas, and J. E. Smith. A Review of Pulse Jet Baghouse and Dual Mechanical Collector Performance on Spreader Stokes Fired Boilers. Paper No. 77-26.4. Presented at the 70th Annual Meeting of the Air Pollution Control Association, Toronto, Canada, June 20-24, 1977.

National Air Pollution Control Administration. Control Techniques For Particulate Air Pollutants Publication Number AP-51, January, 1969.

Oglesby, S., Jr. 1975. Survey Of Information On Fine Particle Control. EPRI 259/Final Report. Southern Research Institute, Birmingham, Alabama. Prepared For Electric Power Research Institute, Palo Alto, California. 247

Oglesby, S. A Review of Technology for Control of Fly Ash Emissions from Coal in Electric Power Generation. Report to Argonne National Laboratory. Southern Research Institute Report No. SORI-EAS-77-243. July 1, 1977. 245 pp.

Parry, E. P. and R. A. Meyer. Gas Flow Measurements in Air Pollution Monitoring. Presented at the 67th Annual Meeting of the Air Pollution Control Association, Denver, CO (June 1974).

Prill, F. W., L. V. Bing, and D. R. Noll. Fabric Filter Applications on Drum Mix Asphalt Plants. Paper Number 77-16-3. 70th Annual Meeting of the Air Pollution Control Association June 20-24, 1977.

Ragland, J. W., K. M. Cushing, J. D. McCain, and W. B. Smith. HP-65 Programmable Pocket Calculator Applied To Air Pollution Measurement Studies: Stationary Sources. U.S. Environmental Protection Agency Report No. EPA-600/2-77-002, October 1976.

Ragland, J. W., K. M. Cushing, J. D. McCain, and W. B. Smith. HP-25 Programmable Pocket Calculator Applied To Air Pollution Measurement Studies: Stationary Sources. Interagency Energy-Environment Research and Development Program Report, EPA-600/7-77-058, June 1977.

Ragland, J. W., D. H. Pontius, and W. B. Smith. 1976. Design, Construct, and Test A Field Usable Prototype System For Sizing Particles Smaller Than 0.5 μm Diameter - Special Summary Report (SORI-EAS-76-094) Southern Research Institute, Birmingham, Alabama. 65 pp.

Ramsey, G. H., et al. EPA Fabric Filtration Studies: 2. Performance of Non-woven Polyester Filter Bags. EPA-600/2-76-168b, U.S. Environmental Protection Agency, June, 1976. 37 pp.

Ranz, W. E., and J. B. Wong. Impaction of Dust and Smoke Particles. Industrial Engineering Chemistry. 44(6):137-138, 1952.

Rao, A. K. Doctoral Thesis, University of Minnesota, Minneapolis, MN, 1975.

Reigel, S. A., and R. P. Bundy. Why the swing to baghouses? Power (January, 1977), pp. 68-73.

## Bibliography (Cont'd.)

Sansone, E. B. and D. A. Weyel. A Note On The Penetration Of A Circular Tube By An Aerosol With A Log-Normal Size Distribution. Aerosol Science, 2, pp. 413-15 (1971).

Schmidt, E. W., J. A. Greseke, and J. M. Allen. Size Distributions Of Fine Particulate Emissions From A Coal-Fired Power Plant. Atmospheric Environment, 10, 1065-1069 (1976).

Schrag, M. P., and L. J. Shannon. Evaluation of Electric Field Fabric Filtration. EPA-600/2-76-041, U.S. Environmental Protection Agency, Washington, D.C., February, 1976. 25 pp.

Seale, L. M. (ed.). Proceedings: Symposium on the Use of Fabric Filters for the Control of Submicron Particulates, April 8-10, 1974, Boston, Massachusetts. EPA-650/2-74-043, U.S. Environmental Protection Agency, Washington, D.C., May, 1974. 316 pp.

Sem, G. J., et al. 1971. State of the Art: 1971. Instrumentation for Measurement of Particulate Emissions From Combustion Sources. Volume I - PB 202665, Volume II PB 202666, Volume III - PB 223393, Volume IV - PB 231919. Thermosystems, Inc., St. Paul, MN. 605 pp.

Sem, G. J. Submicron Particle Size Measurement of Stack Emissions Using The Electrical Mobility Technique. Presented at the Electric Power Research Institute Workshop on Sampling, Analysis, and Monitoring of Stack Emissions, October (1975).

Session 32 - Particulate Control (Filters). Proceedings presented at the 70th Annual Meeting of the Air Pollution Control Association, Toronto, Canada, June 20-24, 1977.

## Bibliography (Cont'd.)

Session 57 - Control Equipment For Particulate Emissions. III - Filtration. Proceedings presented at the 68th Annual Meeting of the Air Pollution Control Association, Boston, Massachusetts, June 15-20, 1975.

Sinclair, D., and G. S. Hoopes. A Continuous Flow Condensation Nuclei Counter. Aerosol Science, 6, pp. 1-7 1975.

Sinclair, D. A Novel Form of Diffusion Battery. Am. Ind. Hygiene Assoc. J., 36(1), pp. 39-42 (1975).

Sinclair, D. A Portable Diffusion Battery. Am. Ind. Hygiene Assoc. J., 33(11), pp. 729-35 (1972).

Sinclair, D., R. J. Countess, B. Y. H. Liu, and D. Y. H. Pui. Experimental Verification of Diffusion Battery Theory. Presented at the 68th Annual Meeting of the Air Pollution Control Association, Boston, MA, 1975. Paper No. 75-41.4.

Smith, W. B., K. M. Cushing, and J. D. McCain. 1974. Particle Sizing Techniques For Control Device Evaluation. EPA-650/2-74-102. U.S. Environmental Protection Agency, Washington, D.C. 127 pages. NTIS-PB 240670/$5.75.

Smith, W. B., and R. R. Wilson, Jr. Development and Laboratory Evaluation of A Five-Stage Cyclone System. EPA-600/7-78-008, January 1978. U.S. Environmental Protection Agency, Research Triangle Park, NC. 66 pp.

Smith, W. B., K. M. Cushing, G. E. Lacey, and J. D. McCain. 1975. Particle Sizing Techniques For Control Device Evaluation. EPA-650/2-74-012a. U.S. Environmental Protection Agency, Washington, D.C. 132 pp. NTIS-PB 245184/$5.75.

## Bibliography (Cont'd.)

Smith, F. H. 1974. The Effects Of Nozzle Design And Sampling Techniques On Aerosol Measurements. EPA-650/2-74-076. U.S. Environmental Protection Agency, Washington, D.C. 89 pp.

Soderholm, S. C. Modification of A Commercial Condensation Nuclei Counter For Steady Flow. Atmospheric Environment, Vol. 10: 657-660, 1976.

Southern Research Institute. A Review of Technology for Control of Fly Ash Emissions From Coal in Electric Power Generation. Report Number SORI-EAS-77-243. Birmingham, AL, July, 1977.

Southern Research Institute. 1975. Proceedings Of The Workshop On Sampling, Analysis, And Monitoring Of Stack Emissions. EPRI Special Report 41. Prepared for the Electric Power Research Institute, Palo Alto, CA. 346 pp.

Spink, L. K. and D. M. Considine. Process Instruments and Controls Handbook. McGraw-Hill, 1957.

Standard Havens, Inc. Baghouses and Evaporative Coolers For Air Pollution Control. Kansas City, MO, 1973. 64 pp.

Stephan, D. G. and G. W. Walsh. Residual Dust Profiles In Air Filtrations. Ing. Eng. Chem., (52), 999 (1960).

Stern, A. C. 1968. Air Pollution. Academic Press, New York, New York. 644 pp.

Strauss, W. Industrial Gas Cleaning. 2nd Edition. Pergamon Press, Oxford, England, 1975. 621 pp.

# Bibliography (Cont'd.)

Thomas, J. W. The Diffusion Battery Method For Aerosol Particle Size Determination. J. Colloid Science, 10, p. 246 (1955).

Twomey, S. The Determination of Aerosol Size Distribution From Diffusional Decay Measurements. J. Franklin Inst., 275, pp. 121-38 (1963).

U.S.-U.S.S.R. Working Group - Stationary Source Air Pollution Control Technology. Proceedings of the Symposium On Control of Fine Particulate Emissions From Industrial Sources. January 15-18, 1974. San Francisco, CA. Published by Southern Research Institute, Birmingham, Alabama.

Whitby, K. T. and B. Y. H. Liu. Generation of Countable Pulses by High Concentrations of Subcountable Sized Particles in the Sensing Volume of Optical Counters. J. Colloid Interface Science, 25: 537-46, 1967.

Yeh, H. C. A Fundamental Study of Aerosol Filtration by Fibrous Filters. Ph.D. Thesis, University of Minnesota, March, 1972.